HGL
HydroGeoLogic, Inc
Exceeding Expectations

Kansas City Operations

March 31, 2017

Ms. Koni Fritz
AES Project Officer
U.S. Environmental Protection Agency, Region 7
11201 Renner Boulevard
Lenexa, KS 66219

RE: Sample Management Plan
Sampling and Analysis Plan
Cherokee County Site – OU8 Railroads, Cherokee County, KS
U.S. EPA Region 7 AES Contract No. EP-S7-05-05;
Task Order No. 0073
EPA Task Order Project Officer: Elizabeth Hagenmaier

Dear Ms. Fritz:

HydroGeoLogic, Inc. (HGL) is pleased to submit one electronic copy of the Site Management Plan and Sampling and Analysis Plan for the Cherokee County Site – OU8 Railroads, Cherokee County, KS. This document was prepared in accordance with Task Order 0073 and our EPA-approved task order proposal dated January 27, 2017.

Should you have any questions or comments, please contact us at 913-317-8860.

Sincerely,

Andrea Fletcher
HGL Task Order Manager

W. Alan Rittgers, P.G.
AES Program Manager

40542334

Enclosures

6340 Glenwood, Suite 200, Building #7, Overland Park, KS 66202
Phone: (913) 317-8860 Fax: (913) 317-8868
www.hgl.com

FINAL
SAMPLING AND ANALYSIS PLAN
REMEDIAL DESIGN
CHEROKEE COUNTY SITE - OU 8 RAILROADS
CHEROKEE COUNTY, KS

Prepared for:

U.S. Environmental Protection Agency Region 7
11201 Renner Boulevard
Lenexa, KS 66219

Architect and Engineering Services Contract EP-S7-05-05
Task Order: 0061

March 2017

HydroGeoLogic, Inc

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CHEROKEE COUNTY SITE - OU8 RAILROADS
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U.S. Environmental Protection Agency Region 7
11201 Renner Boulevard
Lenexa, KS 66219

Prepared by:

HydroGeoLogic, Inc.
6340 Glenwood, Suite 200, Building #7
Overland Park, KS 66202

March 2017

SAMPLING AND ANALYSIS PLAN
REMEDIAL INVESTIGATION
CHEROKEE COUNTY SITE - OU8 RAILROADS
CHEROKEE COUNTY, KANSAS
TASK ORDER 0073

PROJECT: Remedial Design
Cherokee County Site - OU8 Railroads

TASK ORDER NUMBER: 0073

HYDROGEOLOGIC, INC.

PROGRAM MANAGER: W. Alan Rittgers, P.G.
TASK ORDER MANAGER: Andrea Fletcher
REGIONAL QC COORDINATOR: Mary Knowles

U.S. ENVIRONMENTAL PROTECTION AGENCY – REGION 7

TASK ORDER PROJECT OFFICER (TOPO): Elizabeth Hagenmaier

PREPARATION DATE: March 31, 2017
PLAN PREPARER: HGL
REVISION: 0
APPROVED BY:

W. Alan Rittgers, P.G.
HydroGeoLogic, Inc.
Program Manager

Andrea Fletcher
HydroGeoLogic, Inc.
Task Order Manager

Mary Knowles
HydroGeoLogic, Inc.
Regional QC Coordinator

Diane Harris
U.S. Environmental Protection Agency, Region 7
Quality Assurance Officer

Elizabeth Hagenmaier
U.S. Environmental Protection Agency - Region 7
Task Order Project Officer (TOPO)

DOCUMENT DISTRIBUTION LIST:

Elizabeth Hagenmaier, TOPO – 1 copy for use/distribution
Andrea Fletcher, TOM – 1 copy for use/distribution

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LIST OF ACRONYMS AND ABBREVIATIONS

AES	Architect and Engineering Services
ASR	Analytical Services Request
CCR	Cherokee County Site OU8 Railroads
CERCLIS	Comprehensive Environmental Response, Compensation and Liability Information System
CoC	chain of custody
DQO	data quality objective
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
FD	field duplicate
FSP	Field Sampling Plan
GPS	global positioning system
HGL	HydroGeoLogic, Inc.
HSP	Health and Safety Plan
IDW	investigation-derived waste
MS	matrix spike
MSD	matrix spike duplicate
mg/kg	milligrams per kilogram
OU	operable unit
PDF	portable document format
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RD	Remedial Design
RI	Remedial Investigation
ROD	Record of Decision
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SOP	standard operating procedure
XRF	x-ray fluorescence

FINAL
SAMPLING AND ANALYSIS PLAN
REMEDIAL DESIGN
CHEROKEE COUNTY SITE - OU8 RAILROADS
CHEROKEE COUNTY, KANSAS

1.0 INTRODUCTION

HydroGeoLogic, Inc. (HGL) is executing a Remedial Design (RD) at the Cherokee County Site OU8 Railroads (CCR) site located in Cherokee County, Kansas (Figure 1.1). This project is being executed under Region 7 U.S. Environmental Protection Agency (EPA) Architect and Engineering Services (AES) contract EP-S7-05-05, Task Order 0073. This RD is part of the comprehensive investigation and remediation of the overall Cherokee County Superfund Site; Comprehensive Environmental Response, Compensation, and Liability Information System (CERCLIS) identification number KSD980741862. For the purposes of the investigation and remediation of the overall Cherokee County Superfund Site, the site was divided into operable units (OUs): the railbeds included in the CCR RD are identified as OU8.

This Sampling and Analysis Plan (SAP) conveys the sampling approach and procedures for collecting environmental samples in support of the CCR RD. Additional samples were requested to better delineate the contamination within OU8. The RD samples will be collected following the same approach that was used for the Remedial Investigation (RI) sampling. The SAP consists of the Field Sampling Plan (FSP) and the Quality Assurance Project Plan (QAPP). The FSP details the data collection activities planned for the RD activities, field screening and sampling procedures, and sample management. The FSP is augmented by the QAPP documents, which include the Generic QAPP for Region 7 Superfund Lead-Contaminated Sites and the site-specific CCR Site QAPP Addendum appended to the Generic QAPP. The QAPP documents are provided in Appendix A and present the analytical methods, sample handling and custody, and samples required to meet the quality control (QC) requirements of the field effort. The FSP, Generic QAPP, and QAPP Addendum provide specific details regarding the planned sampling scheme, data quality objectives (DQOs) for field and laboratory data, and data management procedures that will be employed to ensure that data collected are of sufficient quantity and known quality to support decision making and risk analysis. All field activities will be conducted in accordance with the health and safety measures detailed in the Health and Safety Plan (HSP) provided in Appendix B.

1.1 PROJECT OBJECTIVES

During the operation of the mines in Cherokee County, private railroads were constructed to join conventional large-scale railroads to the individual mining operations. Chat from surrounding mine waste piles was used to construct the ballasts in the railroad beds. The rail beds are being investigated to determine the extent of metals contamination associated with the mine waste used in their construction. The objectives of the RD for the Cherokee County OU8– Railroads site include:

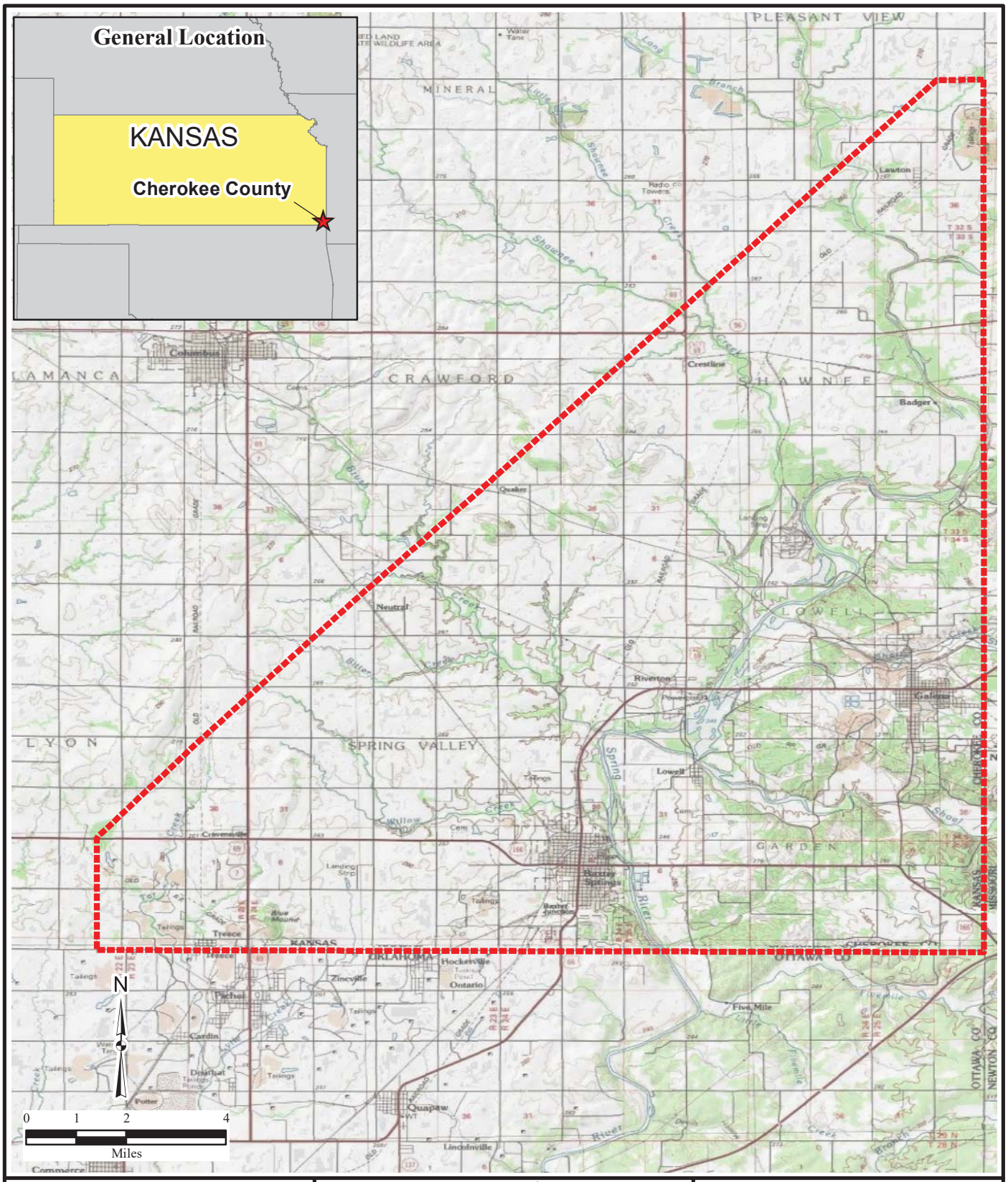
- Characterize lead and zinc contamination in soils along the identified rail lines that were not sampled during the Remedial Investigation (RI); and
- Provide findings from sampling activities as an appendix to the Preliminary Design Report.

1.2 SCOPE OF WORK

The field sampling regime detailed in this SAP has been designed to obtain the requisite data to satisfy the project objectives as listed in Section 1.1. Historical records for the site indicate that, as a result of past Site operations, contaminated chat may have been placed as ballast to construct the former rail lines that transect the county. Past investigations of the mining sites in the region have documented contamination in soil. Specific scope elements for the additional fieldwork that will be conducted to support the Remedial Design are listed below:

- Arrange access with up to 75 property owners for sampling locations county-wide.
- Collect grab samples at up to 60 locations at 6-inch intervals from the surface to a depth of 4 feet using a backhoe. Soil from each interval will be collected from the backhoe bucket, placed in a disposable plastic bag, homogenized and then screened using a field portable x-ray fluorescence (XRF) spectrometer.
- Excavate horizontal cross sections at half (up to 30) of the locations to allow visual determination of how far the ballast extends from the center of the rail bed, and support a determination of the approximate volume of contaminated soil along the rail line.
- Collect confirmation samples for off-site laboratory analysis (fixed-laboratory analysis) to validate the accuracy of the XRF unit. The frequency of confirmation sample collection is 10 percent of the XRF samples. Confirmation soil samples will be collected and submitted to the EPA off-site laboratory for lead and zinc analyses.

FIGURE(S)



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Source: HGL, ESRI Online USA Topo Maps

Legend

Site Boundary

Figure 1.1
Site Vicinity Map

HGL HydroGeoLogic, Inc

2.0 SITE LOCATION AND DESCRIPTION

The Cherokee County Superfund Site spans 115 square miles and represents the Kansas portion of the Tri-State mining district (Figure 1.1). The Tri-State Mining District covers approximately 2,500 square miles in northeast Oklahoma, southwest Missouri and southeast Kansas. The Tri-State Mining District was one of the foremost lead-zinc mining areas of the world and provided nearly continuous production from about 1850 until 1970. During this period, the district produced an estimated 500 million tons of ore, with about 115 million tons produced from the Kansas portion of the district. EPA has formerly listed four mining-related Superfund Sites in the Tri-State Mining District: the Tar Creek Site in Oklahoma; the Jasper County and Newton County sites in Missouri; and the Cherokee County Site in Kansas.

2.1 OPERATIONAL HISTORY

The Tri-State Mining District is characterized by a variety of mine waste features that contain sparse to no vegetation. Local stream systems also contain mining wastes and mining-impacted sediments and surface water. Residential areas are adjacent to mine waste accumulations in some areas or have suffered historic impacts as a result of smelting. Lead and zinc are found in mining wastes and soils at maximum concentrations of several thousand milligrams per kilogram (mg/kg), while cadmium is typically found at levels less than 500 mg/kg.

During the years the mines operated, railroads were constructed in Cherokee County to join conventional large-scale railroads to the individual mining operations. Figure 2.1 illustrates the former rail locations through the County. Historically, the ballast used in the railroad beds was composed of chat from surrounding mine waste piles. Traditionally, these historical railroads were abandoned in place when mining operations ceased at that mine. Currently, the historical rail lines that cross through private property vary in condition from showing little degradation to being unidentifiable as former rail lines. Depending on the current use of the area, some former rail lines exhibit extensive vegetative regrowth with a thick organic layer, while others have been incorporated into the surrounding area.

Recently, many rail lines were abandoned by railroad companies and reverted to the property owner through the Surface Transportation Board. Regional plans exist to convert some historic rail beds to the national Rails to Trails program. This conversion program has begun in the Missouri part of the region with potential expansion into Kansas. Several historical rail lines have been addressed during previous remedial actions on properties where they were encountered and some ballast may have been completely removed as a result of subsequent construction activities, such as highway cuts. With the potential changes in land use, the exposure scenarios have changed. Some segments of the relic rail lines that ran through other OUs at the Cherokee County Site have been investigated and remediated; other portions of the rail lines were investigated during the RI.

2.2 SUMMARY OF PAST INVESTIGATIONS

The Cherokee County Superfund Site was placed on the National Priorities List in 1983. As listed, the Cherokee County Site encompasses 115 square miles including the following nine OUs:

- OU1 – Galena Alternative Water Supply;
- OU2 – Spring River Basin;
- OU3 – Baxter Springs subsite;
- OU4 – Treece subsite;
- OU5 – Galena Groundwater/Surface Water;
- OU6 – Badger, Lawton, Waco, and Crestline subsites;
- OU7 – Galena Residential Soils;
- OU8 – Railroads; and
- OU9 – Tar Creek Watershed.

These nine OUs encompass most of the area where mining occurred within the Site and where physical surface disturbances were evident. The Site consists of mine tailings, soil, sediment, surface water, and groundwater contaminated with heavy metals (principally lead, zinc, and cadmium). The primary sources of contamination at the Site are the residual metals in the abandoned mine workings, chat piles, and tailing impoundments in addition to historical impacts from smelting operations. Numerous remedial and removal actions have taken place throughout the site as noted in Records of Decision (RODs) and Five-Year Reviews for the Site. The additional sampling planned to support the RD is a continuation of the RI investigation of rail lines that are not associated with investigations at areas identified as mining sites.

2.3 ENVIRONMENTAL SETTING

2.3.1 Land Use and Zoning

Previous studies classified recent land use of the Cherokee County area primarily as cropland and pasture with some forest and residential use. Some open land is in use as mine waste repositories associated with the remediation effort in the area. Cultivated crops are primarily wheat and soybeans, with some grain sorghum, corn, barley, oats, and alfalfa. Tall fescue is the main cultivated grass grown for pasture and hay. Beef and dairy cattle are raised in the area as well. There is a coal-fired power plant on the Spring River near Empire Lake and various light industry in and around Baxter Springs. Chat is processed at both the Baxter Springs and Treece subsites, especially at Treece by Bingham Sand and Gravel. Chat from these commercial facilities is loaded into trucks and hauled to various locations in Kansas, Oklahoma, and Missouri.

Most of the surface and mineral rights within the subsites, excluding roads and highways, are privately owned. Corporations own only 6 percent of the surface rights and 7 percent of the severed mineral rights of the area.

2.3.2 Property Ownership

Property ownership within Cherokee County includes private landowners, commercial businesses, railroads, and local and state government. Tax maps showing land ownership are available from the Cherokee County Kansas Appraiser website, http://www.cherokeecountyks.gov/Departments/Appraiser/tabid/12914/Default.aspx.

Additional research to identify the locations and ownership of defunct rail lines is a component of the RD sampling effort and will be completed before sampling activities commence.

2.3.3 Site Topography

The topography in southeast Kansas is generally gently sloping, except in the river valleys and areas of waste stockpiles and collapsed mine areas. Topographic relief in the stockpile areas approaches over 50 feet. Topographic relief associated with existing mine shafts and collapse features is on the order of 50 to 100 feet. Side slopes along the collapse features are generally very steep. It is anticipated that the site topography in the rail road sample locations will follow the regional topography.

2.3.4 Existing Utilities

Existing utilities are generally located within easements that follow the county road system or where residential/industrial service lines are present. Before any intrusive activities are conducted for the RD, Kansas One-Call Service will be notified of excavation activities so that any subsurface utilities in the excavation areas can be demarcated, if present.

2.4 CONTAMINANTS OF INTEREST

The primary contaminants of interest are lead and zinc. Elevated concentrations of lead and zinc have been found throughout the site.

FIGURE(S)

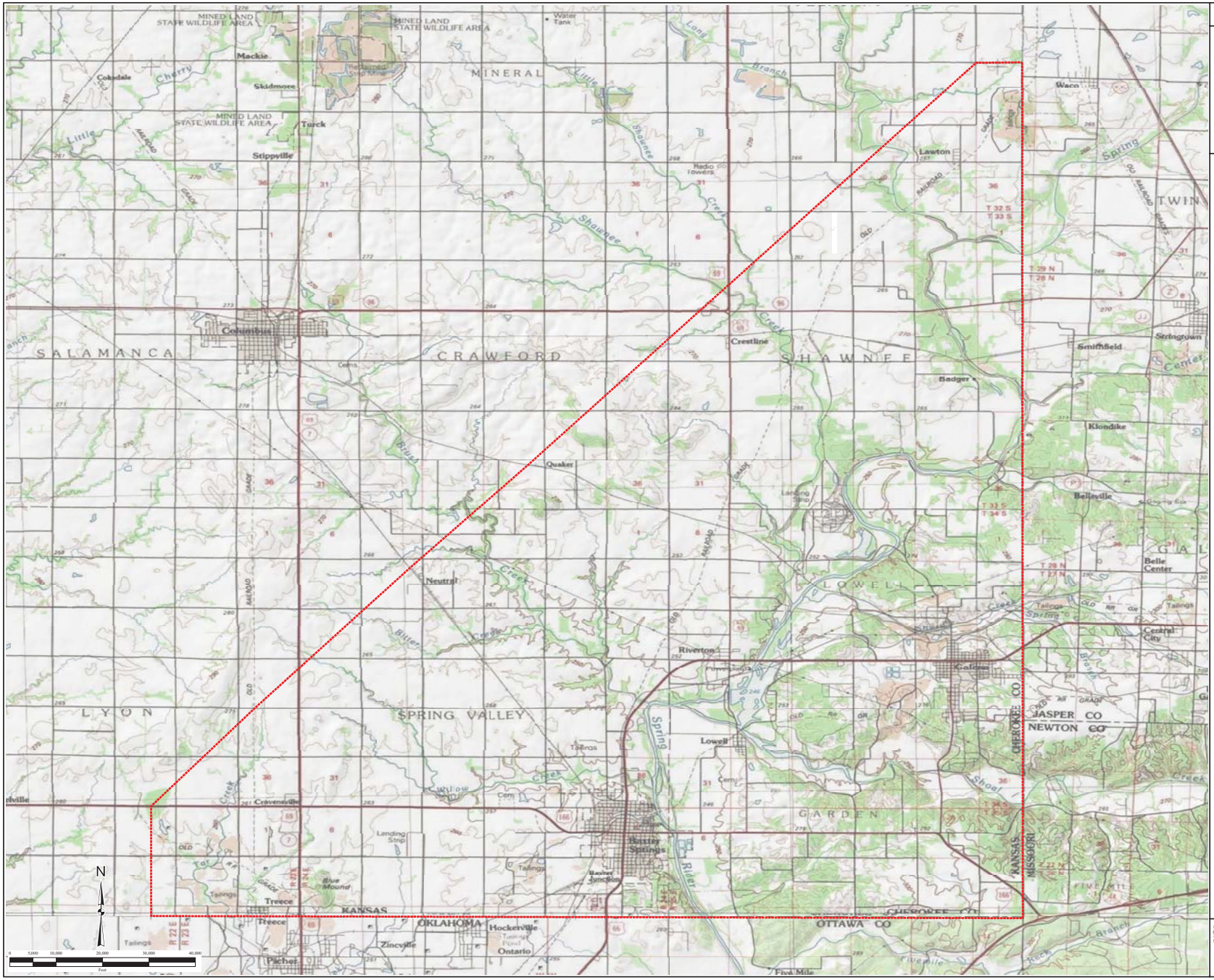

Figure 2.1
Site Features

Legend

Site Boundary

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Source: HGL, ESRI Online USA Topo Map

HGL

3.0 SAMPLING PROGRAM AND ANALYTICAL SUMMARY

This section describes the rationale and procedures for field efforts that will be conducted to meet the objectives defined in Section 1.1. Field activity methods and procedures are discussed in Section 4.0.

3.1 OVERVIEW OF SAMPLING REGIME

The RD activities for this FSP are listed below and detailed in the following subsections:

- Evaluate metals contamination in rail bed surface and subsurface soil using an XRF.
- Collect surface and subsurface soil confirmation samples from rail beds evaluated with the XRF.
- Evaluate metals (lead and zinc) contamination in surface and subsurface soil samples.

These activities will be conducted to establish profiles of metals concentrations of select metals in rail beds not investigated during the RI to achieve the following objectives:

- Define the nature and extent of contamination in former rail beds; and
- Provide data and information to enable EPA to assess risks to the environment based on the risk assessment conducted as part of the RI.

All sampling activities will be performed according to procedures specified in EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (OSWER 9285.7-50) (EPA, 2003) and EPA standard operating procedure (SOP) *Protocols for the Region 7 Lead-Contaminated Residential Yard Soil Cleanup Actions, Procedures and Sequencing,* 4220.03A (Appendix D). All sample analyses will be, at minimum, consistent with those defined under the *Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites*, presented in Appendix A. All confirmation samples submitted to the EPA laboratory will be analyzed for lead and zinc. All environmental and QC samples will be uniquely identified and documented in a sampling team logbook and on field sheets, as described in Section 5.0.

3.2 SOIL INVESTIGATION

Surface and subsurface soil samples from the former rail lines will be collected from approximately 60 locations across 75 properties and analyzed using a combination of XRF screening and fixed-laboratory confirmation analyses. Ten percent of the soil samples analyzed using ex situ XRF will be sent as confirmation samples to the EPA off-site laboratory for lead and zinc analysis. The confirmatory samples will be selected from the lower, middle, and upper range of concentrations measured by the XRF (EPA, 2007).

In addition, appropriate quality assurance (QA)/QC samples also will be prepared and collected for the site including duplicate and matrix spike (MS)/matrix spike duplicate (MSD) samples. Duplicate samples will be collected at a rate of 10 percent of the total number of confirmation samples. MS/MSD samples will be collected at a rate of 20 percent of the total number of confirmation samples. All QC samples will be uniquely identified and will be

documented in the field logbook and on field sheets. All QC samples will be sent to the EPA laboratory for confirmation analysis. The EPA Region 7 Generic QAPP and QAPP Addendum (Appendix A) discuss sample documentation and handling as does Section 5 of this FSP.

The lead concentrations from field XRF screening analysis and the fixed-laboratory confirmation data will be compared to determine whether the data meets the quality goals specified in the QAPP (Appendix A).

The XRF and fixed-laboratory data obtained during the RD field effort results will be compared to the removal action objectives developed by EPA for the site to determine whether further remedial action is warranted.

4.0 FIELD ACTIVITY METHODS AND PROCEDURES

This section provides the rationale and procedures that will be used to conduct the RD field activities for the CCR site. The following is a summary of field activities that will be performed by HGL personnel.

- Conduct Site Visit to determine if rail lines that have not been sampled are accessible and intact;
- Identify locations that were not previously sampled to further delineate contamination;
- Obtain property access;
- Complete mobilization activities
- Collect surface and subsurface rail bed samples
- Manage investigation-derived waste (IDW)

This FSP references EPA SOPs pertinent to the specific sampling event. Referenced SOPs are provided in Appendix D. Referenced field forms are provided in Appendix C. All field activities will be conducted in accordance with the HSP that defines and documents the health and safety procedures to be implemented for the project (Appendix B). This document incorporates by reference the HGL corporate Health and Safety Plan and is inclusive of all activities to be conducted under this task order. The HSP meets the requirements of 29 Code of Federal Regulations 1910 and 1926, National Institute of Occupational Safety and Health Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities (1985), EPA Order 1440.1 - Respiratory Protection, and EPA Order 1440.3 - Health and Safety Requirements for Employees Engaged in Field Activities.

4.1 MOBILIZATION

HGL will identify and provide all necessary personnel, equipment, and materials for mobilization and demobilization to and from the site for the purpose of conducting the RD field activities. Equipment mobilization entails ordering required supplies and sample containers from EPA Region 7 Environmental Services Division and renting or purchasing any additional equipment or supplies needed. Mobilization activities will include coordination with the heavy equipment subcontractor for sampling support.

4.2 PROPOSED CLASSIFICATION SYSTEM

A proposed classification system was developed to establish guidelines that can be extrapolated to all rail lines in Cherokee County during the RI. The classification system consisted of three elements: current condition, distance from source areas, and current/future reuse. After completion of the RI, the classification categories were revised as follows and shown on Figure 4.1:

- Active Line,
- Former Lines Within OU8,
- No Longer Present or Remediated, and
- Addressed Under Other OU.

4.3 PROPOSED SAMPLE LOCATIONS

Proposed sample locations were determined during Site visit conducted March 23, 2017. Figure 4.1 details the proposed sample locations for the RD and cover areas that were not sampled during the RI. Actual sample locations will be updated based on access received.

4.4 SAMPLING METHOD

Test pits will be excavated using a rubber-tired backhoe or equivalent. Each test pit will be excavated in incremental lifts at 6-inch intervals beginning at the surface to a depth of 4 feet below ground surface. Soil from each interval will be collected from the backhoe bucket, placed in a disposable plastic bag for screening using the XRF unit. At approximately half the locations, the lateral extent of the rail line ballast will be assessed by excavating additional areas using a shovel to see if chat is still present. During the RI, it was found that many areas were overgrown or were submerged under water and were not accessible for sampling. The number of horizontal locations will be determined in the field based on the visible ballast. Samples from each horizontal location will also be placed in a disposable plastic bag and screened with the XRF unit. Confirmation samples for laboratory analysis will be collected at a frequency of 10 percent from the material collected and packed into EPA-provided glass sample containers. Each sample location will be recorded using a global positioning system (GPS) unit. The GPS coordinates will be recorded in the field logbook.

4.5 EQUIPMENT, SUPPLIES, AND CONTAINERS

HGL has identified the equipment and supplies necessary to support the field activities. These items are summarized in Table 4.1. The sample containers and associated preservatives for all site sampling activities are summarized in Table 4.2. All sample containers will be pre-cleaned and traceable to the facility that performed the cleaning. Sampling containers will not be cleaned or rinsed in the field.

4.5.1 Property Access

Property access will be obtained through access agreements signed by the property owner. The access agreements will initially be mailed out to each property owner. Follow-up letters will be used as the next step in obtaining access for those properties where owners do not respond to the initial mailing. After two attempts by HGL, EPA will be responsible for contacting property owners to obtain access. Existing access agreements in use during the RI or on other OUs in Cherokee County will also be used on this project.

4.5.2 XRF Field Screening

Screening of the soil for lead concentrations will be accomplished using a portable Niton™ XRF instrument. Ex situ soil XRF field analysis will be conducted in accordance with the guidelines described in the *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003) and EPA SOP #1713, *Portable X-Ray Fluorescence Operating Procedures,* January 1995, provided in Appendix D. The XRF will be calibrated in the field at the

manufacturer's recommended frequency and measurements will be documented in a dedicated field logbook.

Before collecting XRF readings, the method precision for the instrument must be documented on a daily basis. Relative standard deviation (RSD) is used to assess method precision. The RSD must be less than 20 percent for XRF readings to meet the project objectives for precision. If the RSD is greater than 20 percent, the method precision should be rechecked. If the recheck RSD is greater than 20 percent, the manufacturer will be contacted to resolve the issue. The steps listed below will be followed to calculate the method precision.

1. Obtain check samples with varying lead concentrations.
2. Analyze one of the check samples seven times. The samples shall be analyzed until the instrument uncertainty is less than +/- 10 percent of the measured values. Note: The check samples should be alternated daily to avoid completing a precision calculation using the sample analyzed the previous day.
3. Document the instrument readings in the field logbook.
4. Calculate the standard deviation and mean concentration for the seven readings.
5. Divide the standard deviation by the mean concentration.
6. Multiply value obtained from step 5 by 100 to obtain the RSD.
7. Record all readings and calculations in a dedicated field logbook.

4.5.3 XRF Soil Sample Analysis

After calibration verification, XRF analysis of the soil samples for lead, and zinc will be conducted. Sufficient sample volume will be collected to allow for XRF soil screening, as well as for possible EPA laboratory confirmation analysis. Each soil sample will be collected using a clean, stainless-steel spoon, spade, or trowel and placed in a disposable plastic bag. Debris such as sticks and larger stones will be removed. Three readings and uncertainty values will be recorded, averaged, and documented for each interval. Uncertainty values are expressed as a +/- or error value. All three readings must be within 10 percent of each other. If any of the three readings falls outside of the $\pm$ 10 percent range, the sample must be remixed and the readings recollected until the $\pm$ 10 percent criteria is achieved. All XRF screening results at each interval also will be recorded in the field logbook. After all XRF readings have been completed and documented, excess soil not submitted to the laboratory for analysis will be returned to the rail bed. Each sampling location will be restored to the extent possible to its post-excavation condition.

4.6 EQUIPMENT DECONTAMINATION PROCEDURES

Backhoe equipment used to perform soil sampling will undergo gross decontamination between sample locations which will consist of using a brush to remove any visible soil remaining on the bucket. Remaining equipment used to handle soils as part of the field activities will be decontaminated between each location. Decontamination will be performed in accordance with

EPA Region 7 SOP *Sampling Equipment Decontamination,* 4231.2006, provided in Appendix D.

4.7 PERSONAL PROTECTIVE EQUIPMENT

All personal protective equipment to be used for tasks identified as part of the RD is documented in the HSP (Appendix B).

4.8 INVESTIGATION-DERIVED WASTE MANAGEMENT

No IDW is anticipated. Based on the nature of the field activities, no waste requiring special disposal will be generated. Soil collected from the test pits will be returned to the collection location unless it is submitted to the laboratory for analysis. Disposable or expendable materials such as used sampling bags, sampling spoons, and gloves will be combined with general refuse and will be placed in garbage bags for disposal as ordinary household solid waste.

TABLE(S)

Table 4.1
Field Equipment and Supplies
Cherokee County OU8 Railroads Site
Cherokee County, KS

Sampling Supplies	
Sample containers	Deionized water
Shipping material (packaging tape, bubble wrap)	Sampling field forms
Plastic bags	Sample labels
Ice	Chain of Custody forms
Sample shipping coolers	Custody seals
Alconox	Plastic spray bottles
Disposable Scoops	
Sampling Equipment	
Rubber wheel or track mount backhoe	GPS Unit
Contractor/Surveyor Wheels	Niton XRF
Health and Safety	
Nitrile gloves	First aid kits
Hearing protection	Fire extinguishers
Safety Vests	N95 Dust Masks
General Field Operations	
Logbooks	Indelible ink pens
Digital camera	Paper towels
Measuring tape	Trash bags
Batteries – 9 volt, AA	Utility knives
Clear/duct tape	Brushes

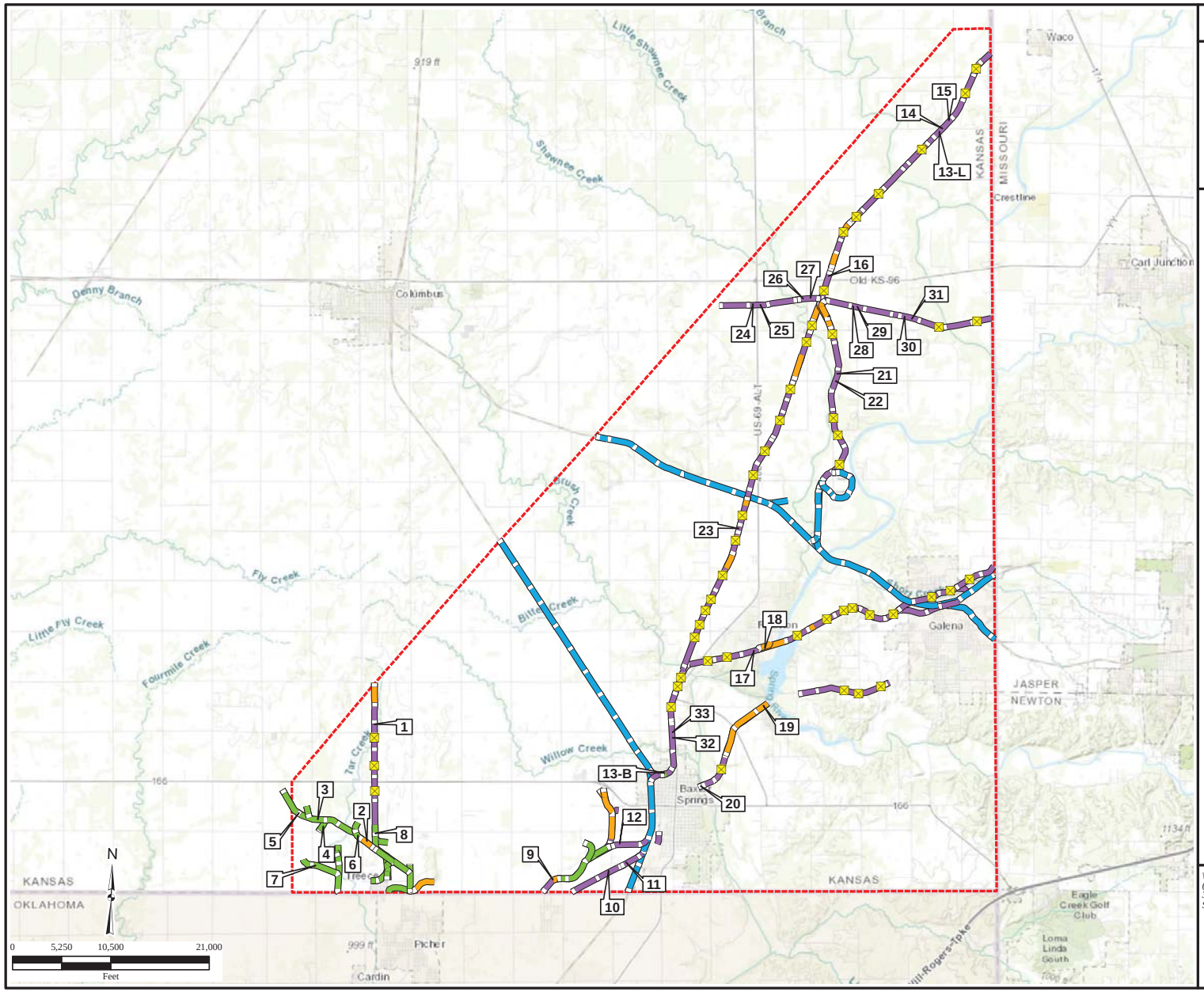
Notes:
GPS = global positioning system
XRF = x-ray fluorescence

Table 4.2
Analytical Methods and Sample Preservation, Holding Time, and Container Requirements
Cherokee County OU8 Railroads Site
Cherokee County, KS

Analytical Parameter	Sample Matrix	Analytical Method	Sample Preservation	Holding Time[1]	Containers
Lead and zinc	Soil	SW-846 6010	None	6 Months	1 - 8 oz. wide-mouth glass jar

Notes:
oz. = ounce
[1] = Holding time is given as number of days prior to extraction / number of days prior to analysis. Dates from day and time of sample collection.

FIGURE(S)



HGL—SAP for Cherokee County Site OU8
Railroads, Cherokee County, Kansas

Figure 4.1
Former Rail Line Classifications and Sample Locations

Legend

- 10 — RI Sample Location
- RD Proposed Sample Location
- Site Boundary

Rail Classification

- Active Line
- Former Lines Within OU8
- No Longer Present or Remediated
- Addressed Under Other OU

Notes:
Rail lines addressed under other OUs were remediated to cleanup levels established for those OUs.

OU=operable unit
RD=remedial design
RI=remedial investigation
SMP=sampling and analysis plan

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3/30/2017 JG
Source: HGL, ArcGIS Online USA Topo Map

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5.0 MANAGEMENT OF FIELD DATA AND SAMPLE TRACKING

5.1 FIELD LOGS AND DATA MANAGEMENT

HGL will document field activities in logbooks, on field forms, and with photographs. Field forms to be used during this project are included in Appendix C. Site conditions and sampling locations will be documented with photographs; the locations, orientation, and subject of each photograph will be recorded in the logbook.

5.1.1 Field Logbooks

Field logbooks will be maintained by the field team in accordance with HGL SOP 4.07 *Use and Maintenance of Field Logbooks,* March 2006 (Appendix D). The logbook documents field activities in sufficient detail to recreate the sampling activities. The logbook will document completion of scheduled activities, and will note problems or deviations from the approved planning documents. Logbooks will be kept in the field team member's possession or in a secure place when not being used. The field team lead will periodically check logbook entries to ensure the required information is recorded as specified in the SOP. At the conclusion of site activities or when the logbook is filled, the logbook will be incorporated into the project file.

5.1.2 Field Forms

In addition to the field logbooks, field forms will be used to record sampling activities and measurements taken in the field. Field forms to be used during RI field activities are provided in Appendix C. Information included on the field sheets will not be repeated in the field logbook. Each completed field sheet will be referenced in the field logbook, as appropriate. Completed field sheets will be provided in the RI Report and maintained in the project file.

5.2 SAMPLE IDENTIFICATION

Each surface soil sample will be uniquely identified as follows:

CCR – SS – 1234

Where:

- SS = surface soil sample
- 1234 = The unique 4-digit CCR sample location number
- 0-6 since this interval will be the same at each location

Each subsurface soil sample will be uniquely identified as follows:

CCR – SO – 1234-XX

Where:

- SO = subsurface soil sample
- 1234 = The unique 4-digit CCR sample location number
- XX = The numerical depth of the subsurface soil sample

In addition, the EPA Region 7 laboratory will pre-assign a unique alpha-numeric identification for each sample collected during the field investigation. The sample numbers will consist of a number designating the Analytical Services Request (ASR) number, and a sequential number for each sample (001, 002, etc.). EPA laboratory personnel will provide preprinted sample labels and sample collection sheets. HGL will include the sample identification number on each sample collection sheet.

Field duplicate QC samples will be identified by an "FD" following the sequential number.

5.3 SAMPLE PACKAGING AND SHIPMENT

Samples will be hand delivered to the EPA laboratory following the sampling event or events if required. If hand delivery is not possible, then samples will be shipped by an overnight delivery service (FedEx or courier) to the EPA laboratory. Because the EPA laboratory only receives samples Monday through Friday, samples will be shipped Monday through Thursday only. When appropriate, a copy of each air bill will be retained and the air bill number will be recorded in a site logbook so the cooler can be easily tracked if mishandled.

5.4 CUSTODY PROCEDURES

Identification and tracking procedures for samples will follow EPA Region 7 SOP 2420.05, *Identification, Documentation, and Tracking of Samples*, September 2010 (Appendix D).

5.4.1 Chain of Custody Requirements

Chain of custody (CoC) procedures will follow the requirements set forth in EPA Region 7 SOP 2420.04, *Field Chain of Custody for Environmental Samples*, April 2012 (Appendix D). The CoC record is employed as physical evidence of sample custody and control. This record system provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. An example CoC record is included with the field forms in Appendix C.

The CoC record is initiated with the acquisition of the samples and remains with the samples at all times. The CoC record includes the name of the field personnel assuming responsibility for the samples and documents transfer of sample custody. As few people as possible should handle the samples during the investigation.

A sample is under custody if one or more of the following criteria are met:

- The sample is in the sampler's possession.
- The sample is within the sampler's view after being in possession.
- The sample was in the sampler's possession and then was locked up to prevent tampering.
- The sample is in a designated secure area.

In addition to the CoC record, custody seals will be used to maintain the custody of samples during shipment. Custody seals are adhesive seals placed on items (such as sample shipping

containers) in such a manner that if the sealed item is opened, the seal would be broken. The CoC seal provides evidence that no sample tampering occurred between shipment of the samples and receipt of the samples by the appropriate laboratory.

Records concerning the cleanliness of empty sample containers, container shipment from the laboratory to the site and security of empty containers at the site will also be maintained in the project file.

5.4.2 CoC Forms for Laboratory Samples

Each sample collected for fixed-laboratory analysis will be recorded on a CoC form by a member of the field sampling team. The field sampler will sign off on the CoC when the samples are relinquished for packaging and shipping of the samples to the laboratories.

The field sampler will sign the CoC when accepting custody of these samples. If the samples are shipped, the sampler will relinquish custody to the courier for shipment by noting "FedEx or courier" and the FedEx air bill number on the CoC form (if applicable). The CoC will be shipped to the appropriate laboratory with the samples, and a copy of the CoC will be maintained by the field sampler.

6.0 DATA MANAGEMENT

The process of data gathering is a coordinated effort and will be conducted by project staff in conjunction with all potential data producers. Both field and fixed analytical laboratory data will be generated for this project. The data management procedures to be used for each type of data are described below.

6.1 FIELD DATA

All field data will be managed in accordance with Section 5.0 Management of Field Data and Sample Tracking. Field measurements/observations will be recorded in a bound field notebook with numbered pages and also on paper forms appropriate for the task at hand (e.g., sample locations, property identification, etc.). Field data will be scanned and given to the HGL project manager who will submit the data to EPA on an as-needed basis. Copies of hardcopy field data will be included in the final RD Report. Original field logbooks, field forms, and other field documentation will be transferred to EPA at the conclusion of the project for incorporation into EPA's permanent project file.

HGL will perform field QA/QC checks and evaluate the XRF field data gathered for usability.

6.2 FIXED LABORATORY DATA

The fixed laboratory data generated during this sampling event will be obtained from the EPA Region 7 off-site laboratory. HGL will receive the data in the form of an electronic data deliverable (EDD) which will consist of Excel, and portable document format (PDF) files of the laboratory deliverables. The EPA Region 7 laboratory will provide data validation on the soil analytical results, and will assign data qualifiers as needed to indicate the data usability. The qualifiers will be placed into the electronic data file. After EPA approves the dataset with the appropriate data qualifiers, the EDD will be released to HGL for reporting.

HGL will review the data validation packages provided by the EPA off-site laboratory. As a part of the data validation review process, the EDDs received from the laboratory will be checked against the hard copy deliverables to ensure accurate transfer of data. In addition, the hard copy will be evaluated for errors in calculation of results.

The EPA Region 7 laboratory will maintain and follow their detailed procedures for laboratory recordkeeping to support the validity of all analytical work. Each data package submitted will contain the laboratory's written certification that the requested analytical method was run and that all QA/QC checks were within established control limits on all samples, with exceptions noted.

6.3 DATA PRESENTATION, INTERPRETATION, AND REVIEW

HGL will present information on the project and the data gathered as an Appendix to the Preliminary Design report. The appendix will include field data collected, along with a summary of XRF screening and laboratory data.

7.0 REFERENCES

U.S. Environmental Protection Agency (EPA), 2003. Superfund Lead-Contaminated Residential Sites Handbook, OSWER 9285.7-5512232we+0. August.

EPA, 2007. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846) Manual, Final Update. February.

APPENDIX A

QUALITY ASSURANCE PROJECT PLAN

Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites

June 2007

Superfund Division
USEPA Region VII
Kansas City, Kansas

APPROVED:

Gene Gunn, Chief
Federal Facilities and Special Emphasis Branch
Date: 6/14/07

Kenneth S. Buchholz, Chief
Enforcement/Fund-Lead Removal Branch
Date: 6/25/07

Scott Hayes, Chief
Emergency Response and Removal Recovery Branch
Date: 7/20/07

Diane Harris, Regional Quality Assurance Manager
Date: 08/31/2007

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APPENDICES

1.0 INTRODUCTION

This generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites was prepared to specifically address Superfund investigations on former and active mining, milling and smelter facilities and the associated impacted areas from the operations of these facilities. This generic Quality Assurance Project Plan (QAPP) is a companion QAPP to the Generic Quality Assurance Project Plan for the Superfund Integrated Site Assessment and Targeted Brownfields Assessment Programs, dated July 2007. The QAPP for the Superfund site assessment and targeted brownfields assessment programs was specific to those programs. This generic QAPP addresses those investigations that are being conducted under the Pre-Remedial (i.e., Site Assessment), Removal and Remedial Programs.

This generic QAPP will assist in the development of site or project specific QAPPs. This should reduce contract costs and promote consistency among all future contractors working on lead site(s). This generic QAPP is a detailed guide for the preparation of site or project specific QAPP, but is not an end all for everything this type of QAPP should and should not contain. A site or project specific QAPP should prescribe the use of specific methods, site specific conditions, and best professional judgment of the site manager.

The Pre-Remedial, Removal and Remedial Programs each have their unique process and ultimate goals in addressing the threat to human health and the environment. The Pre-Remedial Program process is to conduct an initial investigation to determine if threats to the public health and environment actually or potentially exist. During the Pre-Remedial process the human health and ecological threats are identified and a numerical score is obtained to determine the potential for placing the site on the National Priorities List.

The Removal Program process is to conduct investigations to determine if a removal action (i.e., emergency, time-critical or non-time critical) are warranted at a site being investigated. These removal actions can occur during the Pre-Remedial or Remedial phase of the Superfund Process.

The Remedial Program process is to conduct investigations (i.e., Remedial Investigations) for gathering data for the Baseline Risk Assessment for human health and the ecology, and to determine the extent of contamination and to evaluate alternatives for cleanup.

The ultimate success of an environmental data collection effort at Superfund mining, milling and smelter sites contaminated with lead (includes: arsenic, barium, and cadmium) depends on the quality of the data collected and used to make decisions. The Quality Assurance Project Plan (QAPP) is a critical planning document for investigation activities that requires the collection and/or use of environmental data. Thus, the U.S. Environmental Protection Agency's (EPA) policy requires that all environmental data used in decision-making be supported by an Agency-approved QAPP developed from a systematic planning process. The QAPP documents how environmental data collection operations are planned and implemented and how the results are assessed. In addition, the QAPP defines the specific quality assurance (QA) and quality control (QC) activities that will be applied to ensure that the environmental data collected are of the type and quality needed for a specific decision or use.

Current EPA requirements for QAPPs are detailed in EPA's Quality Manual and in EPA Requirements for Quality Assurance Project Plans EPA QA/R-5 (EPA, 2001a). These documents describe the QAPP as divided into four basic element groups covering project management, data generation and acquisition, assessment and oversight, and data validation and usability activities. Each element group is subsequently divided into elements covering different topics; there are 24 elements (Table 1). Not all elements will pertain to every project. Guidance documents for QAPP preparation are available in Guidance for Quality Assurance Project Plans EPA QA/G-5 (EPA, 2002a) and Guidance on Choosing a Sampling

Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan EPA QA/G-5S (EPA, 2002b).

Table 1 List of QAPP Elements

Group A. Project Management	Group B. Data Generation and Acquisition	Group C. Assessment and Oversight
A1 Title and Approval Sheet	B1 Sampling Process Design (Experimental Design)	C1 Assessments and Response Actions
A2 Table of Contents	B2 Sampling Methods	C2 Reports to Management
A3 Distribution List	B3 Sample Handling and Custody	
A4 Project/Task Organization	B4 Analytical Methods	**Group D. Data Validation and Usability**
A5 Problem Definition and Background	B5 Quality Control	D1 Data Review, Verification, and Validation
A6 Project/Task Description	B6 Instrument/Equipment Testing, Inspection, and Maintenance	D2 Verification and Validation Methods
A7 Quality Objectives and Criteria	B7 Instrument/Equipment Calibration and	D3 Reconciliation with User Requirements
A8 Special Training/ Certifications	B8 Inspection/Acceptance of Supplies and Consumables	
A9 Documentation and Records	B9 Non-direct Measurements	
	B10 Data Management	

- ***Project Management*** – These elements address the project history and objectives, and the roles and responsibilities of the participants. These elements ensure that the project goals and approach are clearly understood and that project planning is documented. The Group A project management elements are shown in Table 1 and included in Section 2.0 of this document.

- ***Data Generation and Acquisition*** – These elements describe the measurement system design and implementation and document sampling, analysis, data handling, and QC methods that will be used. The Group B data generation and acquisition elements are shown in Table 1 and included in Section 3.0 of this document.

- ***Assessment and Oversight*** – These elements identify activities for assessing the effectiveness of project implementation and the associated quality assurance and quality control efforts. As such, these elements ensure that the QAPP is implemented as approved. The Group C assessment and oversight elements are shown in Table 1 and included in Section 4.0 of this document.

- ***Data Validation and Usability*** – These elements describe quality assurance activities that occur after data collection or generation. These elements ensure that the data collected conforms to stated acceptance criteria and achieves data quality objectives (DQOs). The Group D data validation and usability elements are shown in Table 1 and included in Section 5.0 of this document.

This Generic QAPP has been developed for Region 7's Superfund Program to be specific to Superfund lead-contaminated sites and is organized in accordance with the 24 QAPP elements specified in EPA QA/R-5 (EPA, 2001a).

The intent of this Generic QAPP for Region 7's Superfund lead-contaminated sites is to provide a framework of procedures for all environmental data collection activities that might occur in accomplishing Site Assessment (SA), Integrated Site Assessment (ISA), Removal Site Evaluation (RES) and Remedial Investigations (RI) activities under authority of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). The Generic QAPP emphasizes the use of proven, validated, and EPA-approved sampling methods and analytical methods such as those in the EPA Contract Laboratory Program Statements of Work and the Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (EPA Publication SW-846) or region specific methods in the EPA Region 7 Environmental Services Division Operations and Quality Assurance Manual (ESDOQAM) (EPA, 2000a). These and other sampling and analytical methods are identified in appropriate sections of this Generic QAPP and will be followed whenever they are sufficient to meet environmental data collection requirements and DQOs.

Task Order (TO) or Procurement Request (PR) are prepared in-house and issued to EPA's contractors to accomplish SA, ISA, RSE and RI projects. All projects conducted by an EPA contractor will have their own site-specific QAPP Addendum or project-specific QAPP. In most instances, the site-specific QAPP Addendum and the Sampling and Analysis Plan (SAP) requirements for a project will be specified within the sampling activities segment(s) of the project work plan(s) along with completing the **Region 7 Superfund Program Addendum for the Generic QAPP for the Superfund Lead-Contaminated Sites Form** (R7 QAPP Addendum Form) as shown in Appendix A. The work plan, containing the elements of a SAP, will be supplemented with appropriate site and sampling location maps and should include a sample summary table or tables that list the sample type (i.e., media and/or purpose), collection methods, and analytical methods to be utilized. Many of the sampling and analytical standard operating procedures (SOPs) that are described in the Region 7 ESDOQAM will be broadly applicable and can be referenced. Additionally, common procedures such as sample handling, chain of custody, data validation, and corrective action should be included **by reference only**. The completed R7 QAPP Addendum Form will be included in the work plan as an attachment or appendix.

There will occasionally be large-scale and/or special projects or pilot studies that may require a "project-specific QAPP" that is independent of this generic QAPP. The requirement to prepare a project-specific QAPP should be identified in the project scope of work and incorporated into the project work plan. The QAPP should be identified in the project scope of work and incorporated into the project work plan. Project-specific QAPPs will be formatted similar to this generic QAPP. If necessary, describe any modifications to the Region 7 ESDOQAM sampling and analytical methods and/or specify any additional data collection procedures that are required to meet the project-specific objectives. Some of the sampling and analytical SOPs that are described in detail within the Region 7 ESDOQAM may broadly applicable and can be referenced. To the extent possible, common procedures such as sample handling, chain of custody, data validation, and corrective action should be included **by reference only**.

2.0 PROJECT MANAGEMENT

2.1 Distribution List

Copies of the approved Generic Quality Assurance Project Plan (QAPP) for Region 7's Superfund Lead-Contaminated Sites activities will be distributed to the following individuals:

EPA Region VII: Gene Gunn, Chief, FFSE
Scott Hayes, Chief, ER&R
Kenneth S. Buchholz, Chief, EFLR
Region 7 National Priority List Coordinator
Site Assessment Mangers who perform SA and ISA activities
On-Scene Coordinators who perform ISA and RES activities
Remedial Project Manager who perform RI activities
Diane Harris, Regional Quality Assurance Manager

Contractor: Program Manager
Quality Assurance Officer
(Contractor will distribute copies within their organization)

2.2 Project and Task Organization

Specific responsibilities of the individuals directly involved with the Superfund's Pre-Remedial, Removal and Remedial Programs are outlined below:

2.2.1 EPA Project Managers

The EPA Project Managers (i.e., Site Assessment Managers (SAMs), On-Scene Coordinators (OSCs), and Remedial Project Managers (RPMs)) will serve as the project manager for the SA, ISA, RES and RI activities. The EPA Project Managers will determine project requirements and ensure that the general scope of work necessary to accomplish the project is provided on the project TO or PR form and/or otherwise communicated to the contractors. The EPA Project Managers will help resolve problems and provide details when necessary to help contractor develop and/or select options for the technical approach and methods to be employed for a project and to develop sampling strategies. The EPA Project Managers will review work plans and cost estimates, and make recommendations to their Branch Chief for any approval/modifications. The EPA Project Managers will perform project oversight by conducting document reviews, audits, site visits, or field oversight activities. The EPA Project Managers will also provide periodic updates to EPA management and/or to EPA Region 7 personnel concerning project status/progress as required.

The EPA Project Managers will oversee all elements associated with the project and will coordinate field activities and other site-related operations with the contractor's Project Manager.

2.2.2 EPA Regional Quality Assurance Manager

The EPA Regional Quality Assurance Manager is required to review and approve this Generic QAPP for Region 7's Superfund Lead-Contaminated Sites and provide general guidance and/or specific instructions to ensure that this Generic QAPP is in compliance with EPA guidance documents and policy. Once the Generic QAPP is revised to meet the standard requirements, it can be coordinated for approval by the EPA Regional Quality Assurance Manager or her designated representatives.

2.2.3 EPA Superfund Branch Chiefs

The EPA Superfund Branch Chiefs will provide overall program management and are the primary decision makers in cooperation with the EPA Region 7 program and/or Project Managers/coordinators. The Branch Chief or his/her designated representative will provide the contractor with the general project scope and objectives and request contractors provide work plans that include site-specific SAP and site-specific QAPP Addendum or project-specific QAPP. The Branch Chief(s) or an appropriate designee will approve recommendations from EPA Project Managers for project work plans and budgets, direct modifications/revisions if required, and ensure that the proper level of management authorization is obtained to approve the project work plan and associated costs (i.e., the project budget). Copies of TO or PR forms, work plans (including cost proposals and SAPs) will be provided to the appropriate EPA Region 7 personnel upon request.

2.2.4 EPA Superfund Site Assessment Coordinator

The EPA Superfund Site Assessment Coordinator monitors the progress of the site assessment projects as well as provides oversight of the Cooperative Agreements funding those projects under CERCLA authority. The EPA Region 7 Superfund Site Assessment Coordinator will review this Generic QAPP and will coordinate necessary revisions between the appropriate EPA Superfund personnel in order to implement the requirements mandated by the Generic QAPP and other QA/QC policy and guidance documents and/or program initiatives.

2.2.5 Contractor Quality Assurance Manager

The contractor's Quality Assurance Manager (QA) Manager for the site-specific project is responsible for monitoring the quality of technical documents generated by the contractor and its subcontractor. He/she will provide direction and guidance to contractor personnel and, through subcontractor QA/project manager(s), to subcontractor personnel performing activities under the contract. The contractor's QA/project manager will maintain a comprehensive quality program based on this Generic QAPP and will issue recommendations about quality to technical staff and management at the contractor's organization. Specific QA/project manager responsibilities include the following:

- Meeting regularly with the contractor's Contract Administrator to review, discuss, and resolve any quality issues and concerns.
- Reviewing, approving, and/or providing guidance to contractor Project Managers and/or technical staff for developing site-specific QAPP Addendums or project-specific QAPPs.
- Interacting with EPA representatives to evaluate the acceptability and qualifications of laboratory and technical subcontractors.
- Conducting field and laboratory audits, identifying nonconformance situations resulting from audits or other QA/QC review activities and notifying the appropriate EPA personnel, contractor's Project Manager(s), the contractor's Contract Administrator and/or regional office manager, and/or subcontractor personnel.
- Providing recommendations and orders for corrective action for all aspects of work that do not meet program standards.
- Facilitating QA problem identification and resolution at both the project- and contract-levels.
- Managing and overseeing all aspects of laboratory procurement and management, data management, data validation, and document generation and review/revision.

2.2.6 Contractor's Contract Administrator

The contractor's Contract Administrator will serve as the primary EPA point of contact for all activities under the particular EPA Environmental Services contract to perform SA, ISA, RSE or RI projects. The contractor's Contract Administrator is ultimately responsible for all field data collection and reporting activities performed in accordance with the QAPP and should ensure that contractor's Project Managers are qualified and provided adequate staff and equipment support to achieve the project requirements. Specific responsibilities of the contractor's Contract Administrator shall include, but may not be limited to the following:

- Receiving, acknowledging, and implementing all TO or PR forms and the resulting approved work plans and other project requirements.
- Designating a Project Manager for each TO or PR.
- Ensuring work plans (including scheduling of work) are submitted for approval by EPA for each TO or PR and for the proper implementation of those approved work plans.
- Providing overall supervision and administrative support to the Projects Manager including providing all the support staff, facilities, administrative capabilities, clerical support and all other resources needed to ensure the successful and efficient accomplishment of TOs or PRs issued and/or project assigned under the contract.
- Reporting and correcting all problems encountered in performing work pursuant to TOs or PRs or in the administration of the contract whether noted by the contractor or noted by representatives of the EPA.
- Preparing and submitting all reports, data, or other deliverables required in the TO or PR forms and ensuring that all deliverables are in compliance with the QA/QC requirements described in the work plan, this Generic QAPP, site-specific QAPP Addendum, or project-specific QAPP documents.

2.2.7 Contractor's Project Managers

The contractor's Project Managers are responsible for implementing all activities identified in the TOs or PRs issued by EPA. The contractor's Project Managers have the authority to commit the resources necessary to meet the technical, financial, and scheduling objectives for the project. The contractor's Project Managers will report directly to the contractor's Contract Administrator and will serve as or provide access information for the major point of contact(s) and control(s) for project-related activities and/or issues. Specific responsibilities of contractor project managers include the following:

- Preparing project work plans with SAP components, site-specific QAPP Addendum or project-specific QAPP for projects involving environmental data collection.
- Verifying that the contractor and/or subcontractor's project team performs contract work and generate contract-related documents and deliverables that comply with all QA requirements in this Generic QAPP and any site-specific QAPP Addendum or project-specific QAPP.
- Monitoring and directing field activities and verifying that appropriate field measurement, field testing, and other field procedures are followed and that appropriate QC checks are conducted.
- Working with the contractor's Quality Assurance Manager and the contractor's Contract Administrator to identify QA problems and to implement effective corrective actions.

On large field investigations the contractor's project manager may be supported by a field team leader (FTL). The FTL is responsible for directing day-to-day field operations and reporting to the contractor's

Project Manager on a daily basis. The FTL will monitor field measurement and sampling procedures to verify the requirements of the work plan documents including site-specific QAPP Addendum or project-specific QAPP are followed. The FTL will also ensure that proper chain-of-custody procedures for sample handling and shipment are utilized. Other specific responsibilities of the FTL include the following:

- Supervising staffing and mobilization activities for field work.
- Overseeing sample collection and field measurements and maintaining field logbook(s).
- Overseeing the activities of all project personnel in the field, including subcontractor personnel.
- Providing the contractor's Project Manager with the required planning, cost and schedule control, records documentation, and data management information related to field activities.
- Facilitating project-level QA/QC problem identification and resolution.

2.2.8 Contractor's Technical Staff

The contractor's technical staff will conduct field activities, gather and analyze data, and prepare various project reports and support materials. The contractor's technical staff will be required to follow procedures and requirements that are specified in TO or PR and approved work plans, QA/QC documents including this Generic QAPP, site-specific QAPP Addendum or project-specific QAPP and other guidance and/or instructions provided by appropriate contractor and/or EPA project/contract management personnel. The contractor's Contract Administrator, with a reasonable amount of assistance from contractor's Project Managers, is responsible for ensuring that all contractor's technical staff members assigned to a project are experienced professionals, who possess the degree of specialization and technical expertise required to effectively and efficiently perform their duties and responsibilities, necessary to complete the required work/task for all TOs or PRs /projects issued under contract.

2.2.9 Team Subcontractor's Project Managers and Staff

Subcontractors may be assigned responsibility for completing all or part of TOs or PRs issued under a contract. On projects with subcontractors having primary involvement, the subcontractor's Project Manager(s) are responsible for the planning, scheduling, budgeting, and reporting related to subcontractor activities. On projects where subcontractors play a supporting role, the subcontractor's Project Manager(s) will coordinate their activities through the prime contractor's Project Manager. Subcontractor's Project Managers will provide technical review of all work conducted by their staff. They will also verify that all work is conducted in compliance with contractor's overall quality requirements and with the quality requirements of any applicable work plans, this Generic QAPP, or site-specific QAPP Addendum or project-specific QAPP.

2.2.10 Team Subcontractor's Quality Assurance Manager

For all portions of the project and data collection activities assigned to the subcontractor component of the team, the team subcontractor's Quality Assurance Manager is responsible for ensuring that all technical services provided by the subcontractor comply with overall EPA contract QA/QC requirements and the project-specific QA requirements of any applicable work plans/SAPs, or site-specific QAPP Addendums or project-specific QAPPs. Specific QA/QC responsibilities of the team subcontractor's Quality Assurance Manager include the following:

- Reviewing and approving work plans/SAPs, site-specific QAPP Addendums or project-specific QAPPs or the applicable segments of such documents under which the subcontractor will provide technical services.
- Monitoring subcontractor performance on the project, including compliance with sample collection, field analysis requirements, sample preparation and analysis methods, sample holding times, required field QC check samples, and data validation as required.
- Maintaining project-specific records of QC data, performance evaluation results, audit comments, and data quality inquiries.
- Applying the subcontractor's QA/QC program to the work done on the project, including reviewing all deliverables before they are submitted to the contractor and verifying that they meet the requirements specified in the project work plan/SAP, or site-specific QAPP Addendum or project-specific QAPP.
- Ensuring that corrective action is implemented when required/directed by appropriate representatives of the prime contractor or appropriate EPA project/contract management personnel.
- Assisting the prime contactor in resolving any QA/QC issues related to the applicable analytical and/or field laboratory's work..
- Facilitating project-level QA/QC problem identification and resolution.

2.3 Problem Definition and Background

This section provides general background information on the EPA Environmental Services contract. In addition, the section outlines the information that should be included in the Problem Definition and Background section of any site-specific QAPP Addendum or project-specific QAPP that would be prepared in response to a TO or PR under the EPA Environmental Services contract.

2.3.1 EPA Environmental Services Contract Background

The EPA utilizes contractors to conduct site assessment, removal assessment and remedial investigation work at sites that are either part of, or considered, potential candidates for Superfund program examination. In addition to Superfund site assessment, other functions may be required to achieve project requirements such as groundwater monitoring evaluation/inspections, technical document reviews and data management activities. All Superfund site assessment, removal site evaluation, and remedial investigation projects are done in accordance with the CERCLA/Superfund pre-remedial, removal and remedial processes. In general, the objectives are to evaluate known or suspected releases of hazardous substances to the environment (i.e., soil, surface water, groundwater air), and to identify the possible sources of the release and the potential impact on likely receptors.

The contractor will be required to furnish all personnel, facilities, equipment, materials, and services necessary for the performance of all work described in the Environmental Services contracts. Specific deliverables and due dates will be as specified in each TO or PR.

2.3.2 Project-Specific Problem Definition and Background

Selection of sites for SA, ISA, RSE or RI sampling, scoring and reporting should utilize any previous investigations or screening activities conducted by the States, EPA or their contractors, or based on other available information collected through desktop/record search activities. The major category of sites

where sampling will be performed includes, but is not limited to active/former lead mining, milling and smelter sites, areas impacted by mining, milling, and smelter activities, mining depositories, transportation routes from mining, milling and smelter sites and the use of mining wastes in public and residential areas.

2.4 Project and Task Description

This is a Generic QAPP for investigations being conducted on EPA Region 7's Superfund lead-contaminated, or potentially contaminated sites under the pre-remedial, removal and remedial programs in the four-state region by EPA and/or its contractors. EPA expects to maintain contracts with firms that provide a wide range of environmental services and who can obtain other specialty environmental-related contract services. Once a site is selected, one of the EPA contractors will be assigned to the project. The site assignment or detection monitoring/sampling activities will be initiated by preparing a TO or PR. The EPA contractor will prepare a site-specific QAPP Addendum or project-specific QAPP which will contain elements of a SAP or a work plan and these documents must clearly identify the proposed sampling, scoring and reporting requirements. Typically, the QA/QC requirements will be provided in the work plan and referencing procedures in, or otherwise using, this Generic QAPP for guidance. The actual site-specific number, location and type of samples will be described in the work plan and its accompanying QA/QC components. Reference to the EPA Generic QAPP requirements will be incorporated in the SAP along with any site-specific QAPP Addendum or project-specific QAPP that may be developed or utilized.

The general objectives of Superfund's site assessment, integrated site assessment, removal site evaluation and remedial investigation sampling efforts are:

- To identify and sample potential source(s) of contamination and thus demonstrate whether a release of hazardous substance has occurred.

- To sample media (Matrix: soil, sediment, soil/rock, groundwater, surface water, dust/wipes and air) that is directly or indirectly exposed/accessible to potential human and/or ecological receptors/targets that may have been impacted or is potentially threatened.

- To estimate the area of contamination and to determine whether the contamination may be attributed to particular actual/potential source area(s).

- To determine whether any contamination (i.e., arsenic, barium, cadmium, cobalt, copper, lead, nickel and zinc) above Maximum Contaminant Levels (MCLs) or health-based benchmarks/action levels exist that poses a threat to human health or the environment.

Soil and groundwater samples will be collected at potential source areas and along suspected migration pathways using direct push sampling probes or conventional drilling when direct push technology will not be able to reach the desired sampling depths. Sediment and surface water samples may also be collected using conventional sampling procedures. Potential targets such as nearby (generally within a mile) private wells and public water supply wells (systems within four miles) may also be sampled.

Sample analysis can either be conducted either in the field or at a lab. Field analysis will be verified by a lab by submission of a percentage of samples depending on site conditions and current methodologies. All critical samples will be analyzed by a lab.

Data collected during the site assessment activities will be used for site scoring and reporting to evaluate whether further regulatory action(s) will be needed at the site or other actual/potential impacted areas off-

site. The contractor and/or subcontractors will provide all site assessment sampling supplies and equipment, unless supplied by EPA.

In general, an initial screening of the site will be performed to determine the site's eligibility for response under CERCLA, assess the need for emergency response activities, determine the potential for non-CERCLA response actions, and ascertain the need to obtain additional information pertaining to the site. If only limited information on site contamination is known, a percentage of samples are typically analyzed for the compounds contained in the Contract Laboratory Program (CLP) Target Analyte List. When information is obtained the analyte list will be tailored to the site. When environmental sampling is planned, a R7 QAPP Addendum Form will be prepared for SA, ISA and RSE activities and will be included in the site-specific QAPP Addendum. This form will be completed in accordance with EPA Region 7 and national program guidance and will encompass the data quality objectives (DQOs) outlined in this Generic QAPP, sampling network design, data collection procedures (including assessment of quality control parameters), special personnel and equipment requirements. The QAPP Form shall be reviewed and approved by the Regional Quality Assurance Manager prior to the start of field work. For RI activities it is not required to complete the R7 QAPP Addendum Form, but the site-specific QAPP shall be reviewed and approved by the Regional Quality Assurance Manager prior to the start of field work. The specific data to be assessed and obtained in additional to the potential sources contamination during the SA, ISA, RSE and RI activities are summarized in Table 2.

TABLE 2
Specific Data

Sample Summary Location	Matrix	Analysis
Residential yards, school yards, parks, daycare centers	Soil	arsenic, barium, cadmium, lead
Stream beds, creeks, ponds, rivers, lagoons, drainage pathways	Soil/Sediment	lead (total)
Borrow sources, rock quarries	Soil/rock	arsenic, barium, cadmium, lead (total) and TCLP-arsenic, barium, cadmium and lead
Municipal Wells	Water	arsenic, barium, cadmium, lead (total and dissolved)
Private Wells	Water	arsenic, barium, cadmium, lead (total and dissolved)
Surface Water	Water	arsenic, barium, cadmium, lead (total)
Interior of private residences (if conditions warrant)	Dust/Wipes	lead (total)
Areas impacted by disturbed contaminated soils, downwind of the site (if conditions warrant)	Air	lead (total)
QC Samples		
Field duplicates	Soil	arsenic, barium, cadmium, lead (total)
Field blanks	Water	arsenic, barium, cadmium, lead (total)
Field duplicates (municipal and private wells)	Water	arsenic, barium, cadmium, lead (lead (total and dissolved)

2.5 Quality Objectives and Criteria for Measurement Data

This section describes quality specifications at two levels: (1) at the level of the decision or study question, and (2) at the level of the measurements used to support the decision or study questions. EPA has developed the Data Quality Objectives (DQO) Process as the Agency's recommended planning process when environmental data are used to select between two alternatives or derive an estimate of contamination. EPA's DQO process is a systematic planning tool designed to ensure that the type,

quantity, and quality of measurement data collected are the most appropriate for supporting decisions that will be based on that data. The DQO process will be used, either formally or informally, for all data collection activities conducted under the EPA Environmental Services contracts to provide the most cost-effective use of program resources. This section describes how the contractor will apply EPA's DQO process to determine the type of data required and presents specific QA objectives for measurement data.

2.5.1 Data Quality Objectives Process

The DQO Process is used to develop performance and acceptance criteria (or data quality objectives) that clarify study objective, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions. The EPA document, Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G-4 (EPA, 2006a), provides a standard working tool for project managers and planners to develop DQO for determining the type, quantity, and quality of data needed to reach defensible decisions or make credible estimates. It replaces EPA's August 2000 document, Guidance for the Data Quality Objectives Process (EPA QA/G-4), that considered decision-making only.

The EPA document, Systematic Planning: A Case Study for Hazardous Waste Investigations EPA QA/CS-1 (EPA, 2006b) shows the use of the DQO Process in the form of a case study. For projects that require data collection, the contractor will follow EPA's DQO process as described in the above guidance documents.

The EPA document, Superfund Lead-Contaminated Residential Sites Handbook, OSWER 9285.7-50 (EPA, 2003), was developed by the EPA to promote a nationally consistent decision-making process for assessing and managing risks associated with lead-contaminated residential sites across the country.

The EPA document, Guidance Manual for the IEUBK Model for Lead in Children, OSWER 9285.7-15-1 (EPA, 1994b) has been developed to assist the user in providing appropriate input to the Integrated Exposure Uptake Biokinetic IEUBK Model for Lead. The IEUBK Model is designed to model exposure form lead in air, water, soil, dust, diet, and paint and other sources with pharmacokinetic modeling to predict blood lead levels in children 6 months to 7 years.

The DQO Process is used to establish performance or acceptance criteria, which serve as the basis for designing a plan for collecting data of sufficient quality and quantity to support the goals of a study. The DQO Process consists of seven iterative steps that are documented in Figure 1. While the interaction of these steps is portrayed in Figure 1 in a sequential fashion, the iterative nature of the DQO Process allows one or more of these steps to be revisited as more information on the problem is obtained.

Each step of the DQO Process defines criteria that will be used to establish the final data collection design. The first five steps are primarily focused on identifying qualitative criteria, such as:

- the nature of the problem that has initiated the study and a conceptual model of the environmental hazard to be investigated;
- the decisions or estimates that need to be made and the order of priority for resolving them;
- the type of data needed; and
- an analytic approach or decision rule that defines the logic for how the data will be used to draw conclusions from the study findings.

The sixth step establishes acceptable quantitative criteria on the quality and quantity of the data to be collected, relative to the ultimate use of the data. These criteria are known as performance or acceptance criteria, or DQOs. For decision problems, the DQOs are typically expressed as tolerable limits on the probability or chance (risk) of the collected data leading you to making an erroneous decision. For estimation problems, the DQOs are typically expressed in terms of acceptable uncertainty (e.g., width of an uncertainty band or interval) associated with a point estimate at a desired level of statistical confidence.

In the seventh step of the DQO Process, a data collection design is developed that will generate data meeting the quantitative and qualitative criteria specified at the end of Step 6. A data collection design specifies the type, number, location, and physical quantity of samples and data, as well as the QA and QC activities that will ensure that sampling design and measurement errors are managed sufficiently to meet the performance or acceptance criteria specified in the DQOs. The outputs of the DQO Process are used to develop a QA Project Plan and for performing Data Quality Assessment.

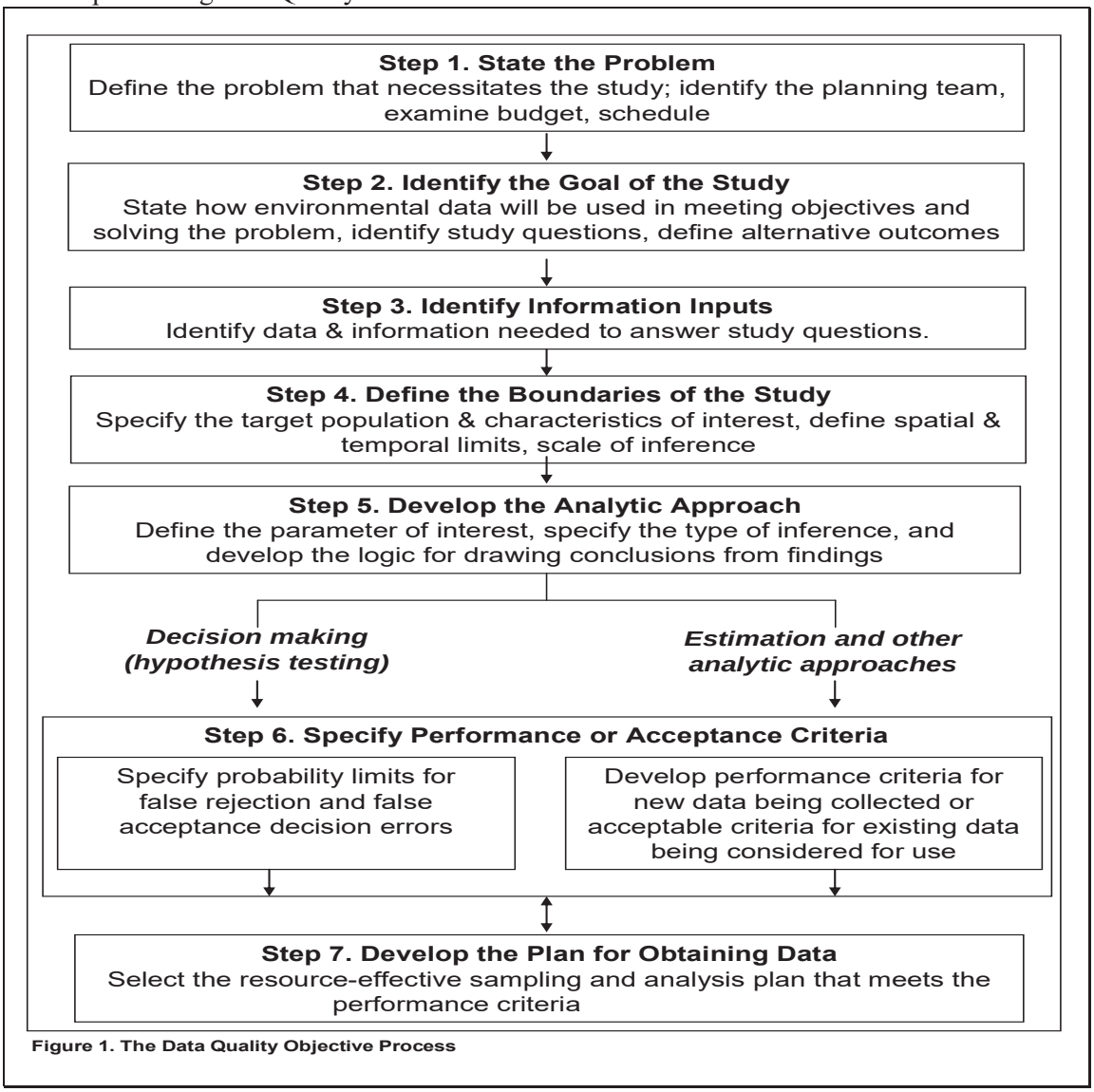


Figure 1. The Data Quality Objective Process

All seven steps of the DQO process may not be applicable to all environmental data collection activities. Examples include activities where specific decisions cannot be identified or studies that are exploratory in

nature. In these situations, the contractor will use the steps of the DQO process that are applicable to help plan the data collection effort.

The DQO process is not complete without a final evaluation, after sample collection and analysis has been completed, of whether DQOs were met. This evaluation, called data quality assessment (DQA), is described in Section 5.3 of this Generic QAPP.

2.5.2 Data Quality Objectives and Criteria for Measurement Data

The **Pre-Remedial, Removal and Remedial Programs** each have their unique process and ultimate goals in addressing the threat to human health and the environment. The **Pre-Remedial Program** process is to conduct an initial investigation to determine if threats to the public health and environment actually or potentially exist. During the Pre-Remedial process the human health and ecological threats are identified and a numerical score is obtained to determine the potential for placing the site on the National Priorities List. The **Removal Program** process is to conduct investigations to determine if a removal action (i.e., time-critical or non-time critical) are warranted at a site being investigated. These removal actions can occur during the Pre-Remedial or Remedial phase of the Superfund Process. The **Remedial Program** process is to conduct investigations (i.e., Remedial Investigations) for gathering data for the Baseline Risk Assessment for human health and the ecology, and to determine the extent of contamination. The ultimate success of an environmental data collection effort at Superfund mining, milling and smelter sites contaminated with lead (includes: arsenic, barium, and cadmium) depends on the quality of the data collected and used to make decisions.

2.5.2.1 Pre-Remedial Program

The Pre-Remedial Program process is to conduct an initial investigation to determine if threats to the public health and environment actually or potentially exist. During the Pre-Remedial process the human health and ecological threats are identified and a numerical score is obtained to determine the potential for placing the site on the National Priorities List.

The sampling activities that are emphasized in the Pre-Remedial Program are the soil, surface water, groundwater, and private and public wells.

2.5.2.1.1 Measurement Objectives for the Pre-Remedial Program

A general description of and rationale for sampling design and field procedures for Pre-Remedial Program Process is provided below:

***Soil Sampling*:** The purpose of soil sampling is to determine if the soils provide a threat to human health and the environment due to the presence of metals and other contaminants. The primary objectives of soil sampling is to determine if threats to the public health and environment actually or potentially exist from tailings or other waste sources in soils downgradient or down-wind from known waste sources and in residential yards (including rights-of way, alleyways, unpaved roads, street easements, and drainage ways) and to provide data to use in developing Hazard Ranking Packages.

Soil samples will be collected near waste areas and in floodplains where stream flows may have deposited tailings, chat, or other waste materials during flood events which over topped the immediate channel banks. Soil samples will also be collected from residential yards, public areas, alleys, street easements, road rights-of-way and drainage ways and surrounding areas. Samples will be discrete samples of surface soils taken at 0 to 1 inches. This depth is necessary to evaluate both the surface horizons for human health and ecological receptors.

The Superfund Lead-Contaminated Residential Sites Handbook, (EPA, 2003) should be consulted for further information on sampling design.

***Waste sampling*:** Various types of waste materials are related to former mining and mineral processing activities. Waste sampling should target areas of known waste material and identified areas. The purpose of sampling these wastes is to determine if threats to the public health and environment actually or potentially exist. In general, tailings and chat areas should be sampled at 0-4 foot, 4-8 foot and continue at 4-foot intervals to depth using Direct Push Technology (DPT). Composite sampling using shovels should be used at any identified waste rock dumps.

***Surface water quality sampling of creeks, rivers and ponds*:** The purpose of surface water quality sampling is to determine if contaminants, primarily total and dissolved metals, in the suspended water column provide a threat to human health and the environment. The primary objectives of water quality sampling are to determine if threats to the public health and environment actually or potentially exist.

***Sediment sampling of streams, rivers and ponds*:** The purpose of sediment sampling is to determine if threats to the public health and environment actually or potentially exist from the various sediments and potential metal flocculants in stream, river, and pond substrates.

Fine sediment fractions are more easily transported and often contain metal complexes and flocculants that are more bioavailable to receptors than coarse fractions. The larger size fractions of many area wastes, particularly chat, are not transported as easily as fine fractions and are not as available to be ingested or adsorbed by humans or aquatic organisms. All sediment samples will be collected and screened to separate the sample into two size fractions for chemical and risk analyses. Samples should be sieved using a Number 35 sieve to separate sediment into size fractions greater than, and less than 0.5 millimeters (mm). This size (0.5 mm) will separate samples into fractions greater than coarse sands and less than medium to fine sands. Evaluating data from both of these size fractions will allow several inferences to be made regarding the nature and extent and transport of waste materials and the risk associated with the different size materials.

***Groundwater sampling*:** The purpose of sampling groundwater is to determine if threats to the public health and environment actually or potentially exist. The primary objectives of sampling groundwater from (other than drinking water wells) are to: (1) determine the water quality and potential exposure levels to human health in areas where residential drinking water wells could be affected by mining activities; and (2) determine groundwater quality in areas associated with mining operations and mining wastes. These objectives will be met by measuring the groundwater potentiometric surface and evaluating observed gradients.

***Residential drinking water well sampling*:** The purpose of sampling potable water from residential drinking water wells is to determine if threats to the public health and environment actually or potentially exist. The primary objectives of sampling potable water from residential drinking water wells is to: (1) determine the water quality and potential exposure levels to human health in residential drinking water wells affected by mining activities; and (2) determine groundwater quality associated with mining operations and mining wastes.

The collection of drinking water samples will follow SOP 4230.1A,"Drinking Water Sample Collection". In addition, two unfiltered drinking water samples will be collected. One sample will be from the well head and the other sample will be taken at the tap or the faucet.

2.5.2.2 Removal Program

The Removal Program process is to conduct investigations to determine if a removal action (i.e., emergency, time-critical or non-time critical) are warranted at a site being investigated. These investigations are called removal site evaluation and are based on whether site conditions meet National Contingency Plan (NCP) criteria for a removal action.

The sampling activities that are emphasized in the Removal Program are the soil, sediment/surface water, interior lead-based paint/interior dust, exterior lead-based paint, air monitoring and private wells.

2.5.2.2.1 Measurement Objectives for the Removal Program

A general description of and rationale for sampling design and field procedures for Removal Program Process is provided below:

***Soil Sampling*:** The purpose of soil sampling is to determine if the soils provide a threat to human health and the environment due to the presence of metals and other contaminants, and identify the extent that this contamination may impact removal decision-making. The primary objectives of soil sampling are to: (1) characterize the nature and extent of contamination by tailings or other waste sources in soils downgradient or down-wind from known waste sources and in residential yards (including rights-of way, alleyways, unpaved roads, street easements, and drainage ways); (2) provide information to allow the risks to human health from exposure to contamination in these areas to be evaluated; (3) determine the chemical stressors that may affect vegetation establishment and/or risk to other ecological receptors; (4) determine whether the level of soil contamination qualifies for removal action; and (5) determine the soil lead levels at the base of the excavation after soil removal.

Soil samples will be collected near waste areas and in floodplains where stream flows may have deposited tailings, chat, or other waste materials during flood events which over topped the immediate channel banks. Soil samples will also be collected from residential yards, public areas, alleys, street easements, road rights-of-way and drainage ways and surrounding areas. Characterization samples will be a combination of discrete and composite samples of surface soils taken from the upper portion of the 0 to 1 inches depth. This depth is necessary to evaluate both the surface horizons for human health and ecological receptors, and the subsurface root-zone to determine the limitations and potential toxicity to plants and soil organisms. The samples should also be properly sieved to determine the metals concentration in the fine fraction of the surface soils Composites should consist of aliquots collected from the same depth. More details on yard soil sampling design can be found in the Superfund Lead-Contaminated Residential Sites Handbook, (EPA, 2003).

***Waste sampling*:** Various types of waste materials are related to former mining and mineral processing activities. Waste sampling should target areas of known waste material and identified areas. The primary objectives of sampling these wastes is to: (1) determine and characterize their bulk geochemistry; (2) characterize the extent of the waste areas; (3) measure the potential for acid generation within the waste rock dumps at the mine area and within the tailing piles; (4) delineate the fate and transport of wastes from wind and water erosion from the main source; and (5) characterize potential exposure concentrations to human and ecological receptors. In general, tailings and chat areas should be sampled at 0-4 foot, 4-8 foot and continue at 4-foot intervals to depth using DPT. Composite sampling using shovels should be used at any identified waste rock dumps.

***Surface water quality sampling of creeks, rivers and ponds*:** The purpose of surface water quality sampling is to determine if contaminants, primarily total and dissolved metals, in the suspended water column provide a threat to human health and the environment. The primary objectives of water quality sampling are to: (1) characterize the water quality and potential exposure potential to human and ecological

receptors due to contact with or consumption of that water; (2) evaluate the geochemistry of the water to determine potential relationships between dissolved and total metal species, as well as determine any significant metal sorption and desorption relationships that could exist between channel substrate sediments and the water column; and (3) determine the extent and degree of transport of contaminants downstream and exposure effects to human health and ecological receptors associated with that transport.

***Sediment sampling of streams, rivers and ponds*:** The primary objectives of sediment sampling are to: (1) define the quality of various sediments and potential metal flocculants in stream, river, and pond substrates; (2) determine potential interactions between substrate sediments and surface water quality near the water-sediment interface and the overall water column; and (3) determine potential exposure concentrations to human health and ecological receptors to coarse and fine sediment fractions.

Fine sediment fractions are more easily transported and often contain metal complexes and flocculants that are more bioavailable to receptors than coarse fractions. The larger size fractions of many area wastes, particularly chat, are not transported as easily as fine fractions and are not as available to be ingested or adsorbed by humans or aquatic organisms. All sediment samples will be collected and screened to separate the sample into two size fractions for chemical and risk analyses. Samples should be sieved using a Number 35 sieve to separate sediment into size fractions greater than, and less than 0.5 millimeters (mm). This size (0.5 mm) will separate samples into fractions greater than coarse sands and less than medium to fine sands. Evaluating data from both of these size fractions will allow several inferences to be made regarding the nature and extent and transport of waste materials and the risk associated with the different size materials.

***Residential drinking water well sampling*:** The primary objectives of sampling potable water from residential drinking water wells is to determine the water quality exposure levels to humans so that potential health risks posed by potable water in residential drinking water wells can be evaluated.

The collection of drinking water samples will follow SOP 4230.1A,"Drinking Water Sample Collection". In addition, two unfiltered drinking water samples will be collected. One sample will be from the well head and the other sample will be taken at the tap or the faucet.

***Indoor dust and lead-based paint sampling*:** The primary objectives for the collection and analysis of dust samples and analysis of the interior paint in residential homes are to: (1) determine the extent of lead contamination in dust from residential homes; (2) determine whether the homes that are sampled for dust also contain lead-based paint; and (3) to collect data that can be included in the IEUBK model (EPA, 1994b and EPA, 2002c) that will be used to prepare the human health risk assessment.

2.5.2.3 Remedial Program

The Remedial Program process is to conduct investigations (i.e., Remedial Investigations) for gathering data for the Baseline Risk Assessment for human health and the ecology, and to determine the extent of contamination.

The sampling activities that are emphasized in the Remedial Program are the soil, sediment, surface water, mine wastes, groundwater, interior lead-based paint/interior dust, exterior lead-based paint, air monitoring and private wells.

The overall quality assurance objective for the remedial investigations for most sites is to develop and implement procedures for field sampling, chain-of-custody (COC), laboratory analysis, and reporting that provide technically and legally defensible results to be used to delineate the nature and extent of contamination, evaluate contaminant migration, assess ecological and human health risk, and support the remedial decision-making process.

2.5.2.3.1 Measurement Objectives for the Remedial Program

A general description of and rationale for sampling design and field procedures for Remedial Program Process is provided below:

***Soil Sampling*:** The purpose of soil sampling is to determine if the soils present a threat to human health and the environment due to the presence of metals and other contaminants, and identify the extent that this contamination may impact remedial decision-making. The primary objectives of soil sampling are to: (1) characterize the nature and extent of contamination by tailings or other waste sources in soils downgradient or down-wind from known waste sources and in residential yards (including rights-of way, alleyways, unpaved roads, street easements, and drainage ways); (2) provide information to allow the risks to human health from exposure to contamination in these areas to be evaluated; (3) determine the chemical stressors that may affect vegetation establishment and/or risk to other ecological receptors; (4) determine whether the level of soil contamination qualifies for removal action; (5) determine the soil lead levels at the base of the excavation after soil removal; and (6) data to support the IEUBK Model (EPA, 1994b and EPA, 2002c).

Soil samples will be collected near waste areas and in floodplains where stream flows may have deposited tailings, chat, or other waste materials during flood events which over topped the immediate channel banks. Soil samples will also be collected from residential yards, public areas, alleys, street easements, road rights-of-way and drainage ways and surrounding areas. Characterization samples will be a combination of discrete and composite samples of surface soils taken from the upper portion of the 0 to 1 inches depth. This depth is necessary to evaluate both the surface horizons for human health and ecological receptors, and the subsurface root-zone to determine the limitations and potential toxicity to plants and soil organisms. The samples should also be properly sieved to determine the metals concentration in the fine fraction of the surface soils Composites should consist of aliquots collected from the same depth. More details on yard soil sampling design can be found in the Superfund Lead-Contaminated Residential Sites Handbook, (EPA, 2003).

***Waste sampling*:** Various types of waste materials are related to former mining and mineral processing activities. Waste sampling should target areas of known waste material and identified areas. The primary objectives of sampling these wastes is to: (1) determine and characterize their bulk geochemistry; (2) characterize the extent of the waste areas; (3) measure the potential for acid generation within the waste rock dumps at the mine area and within the tailing piles; (4) delineate the fate and transport of wastes from wind and water erosion from the main source; and (5) characterize potential exposure concentrations to human and ecological receptors. In general, tailings and chat areas should be sampled at 0-4 foot, 4-8 foot and continue at 4-foot intervals to depth using DPT. Composite sampling using shovels should be used at any identified waste rock dumps.

***Surface water quality sampling of creeks, rivers and ponds*:** The purpose of surface water quality sampling is to determine if contaminants, primarily total and dissolved metals, in the suspended water column provide a threat to human health and the environment. The primary objectives of water quality sampling are to: (1) characterize the water quality and potential exposure potential to human and ecological receptors due to contact with or consumption of that water; (2) evaluate the geochemistry of the water to determine potential relationships between dissolved and total metal species, as well as determine any significant metal sorption and desorption relationships that could exist between channel substrate sediments and the water column; and (3) determine the extent and degree of transport of contaminants downstream and exposure effects to human health and ecological receptors associated with that transport.

***Sediment sampling of streams, rivers and ponds*:** The primary objectives of sediment sampling are to: (1) define the quality of various sediments and potential metal flocculants in stream, river, and pond

substrates; (2) determine potential interactions between substrate sediments and surface water quality near the water-sediment interface and the overall water column; and (3) determine potential exposure concentrations to human health and ecological receptors to coarse and fine sediment fractions. Fine sediment fractions are more easily transported and often contain metal complexes and flocculants that are more bioavailable to receptors than coarse fractions. The larger size fractions of many area wastes, particularly chat, are not transported as easily as fine fractions and are not as available to be ingested or adsorbed by humans or aquatic organisms. All sediment samples will be collected and screened to separate the sample into two size fractions for chemical and risk analyses. Samples should be sieved using a Number 35 sieve to separate sediment into size fractions greater than, and less than 0.5 millimeters (mm). This size (0.5 mm) will separate samples into fractions greater than coarse sands and less than medium to fine sands. Evaluating data from both of these size fractions will allow several inferences to be made regarding the nature and extent and transport of waste materials and the risk associated with the different size materials.

***Groundwater sampling*:** The primary objectives of sampling groundwater (other than drinking water wells are to: (1) determine the water quality and potential exposure levels to human health in areas where residential drinking water wells could be affected by mining activities; (2) determine groundwater quality in areas associated with mining operations and mining wastes; (3) determine the nature and extent of contamination in groundwater from mining activities and wastes; and (4) evaluate potential impacts from the transport of contaminated groundwater to surface water sources or other area potable wells. These objectives will be met by measuring the groundwater potentiometric surface and evaluating observed gradients.

***Residential drinking water well sampling*:** The primary objectives of sampling potable water from residential drinking water wells are to: (1) determine the water quality and potential exposure levels to humans so that potential health risks posed by potable water in residential drinking water wells can be evaluated; and (2) further determine the nature and extent of contamination in groundwater in areas associated with mining operations and mine wastes.

The collection of drinking water samples will follow SOP 4230.1A,"Drinking Water Sample Collection". In addition, two unfiltered drinking water samples will be collected. One sample will be from the well head and the other sample will be taken at the tap or the faucet.

***Indoor dust and lead-based paint sampling*:** The primary objectives for the collection and analysis of dust samples and analysis of the interior paint in residential homes are to: (1) determine the extent of lead contamination in dust from residential homes; (2) determine whether the homes that are sampled for dust also contain lead-based paint; and (3) to collect data that can be included in the IEUBK model (EPA, 1994b and EPA, 2002c) that will be used to prepare the human health risk assessment.

Bioaccessability/bioavailability sampling: The primary objective for collection and analysis of soil and dust sample to measure bioaccessability/bioavailability is to provide site specific input parameters to the IEUBK model (EPA, 1994b and EPA, 2002c) in determining site risk and PRGs. Samples for these analyses should be collected in the same manner as the soil and dust samples above.

Speciation/Apportionment sampling: The primary objective of this sampling and analysis of soil, dust, paint, and other sources of metals contamination is to determine the primary sources of the metals contamination in the soil and dust at residential properties. This analysis is also useful in developing the soil to dust ratio for input to the IEUBK model (EPA, 1994b and EPA, 2002c).

2.5.3 Quality Assurance Objectives for Measurement Data

The project data quality objective is to provide valid data of known and documented quality to determine the levels of lead contamination for comparison to benchmarks. Quality assurance (QA) objectives are usually discussed in terms of accuracy, precision, sensitivity, completeness, representativeness and comparability. Sample collection and field measurement activities will be performed based on SOPs discussed throughout Section 3.0. Analytical results for laboratory blanks, duplicates and QC samples, as well as field blanks and field duplicates will be evaluated to determine bias and representativeness.

The overall QA objective for the EPA contract is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and data reporting that will provide results that will facilitate sound decision-making to protect human health and the environment, support regulatory findings, and that are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this Generic QAPP. The purpose of this section is to address the level of QC effort and the specific QA objectives for sensitivity, accuracy, precision, representativeness, completeness, and comparability of data.

Because of the general nature of this Generic QAPP, it is not possible to provide specific quantitative QA objectives for each environmental measurement and each type of sample matrix. In addition, these QA objectives will depend on the results of the DQO process. However, each project-specific QAPP or work plan with site-specific QAPP Addendum will identify the matrices to be sampled, the numbers of samples that will be collected, and the types of field and laboratory measurements that will be applied to the samples. For each sample matrix and environmental measurement type, the project QA/QC document will specify QA objectives in terms of the following information: types of QC samples and measurements involved, frequency of collection and analysis of QC samples and measurements, how the QA objective is measured, the acceptance criteria or QC limits for that measurement, and corrective action to be taken when a QC limit is not met.

All analytical data will be evaluated for compliance with QC limits. Typically, when analytical data do not meet the QC limits, corrective action might be initiated and the data might be qualified or rejected. Corrective action may include stopping the analysis; examining instrument performance, sample preparation, and analysis information; recalibrating instruments; re-preparing and reanalyzing samples; and informing the contractor's Contract Administrator, contractor's Quality Assurance Manager, and the contractor's Project Manager of the problem.

The following subsections address the level of QC effort and general objectives for sensitivity; accuracy and precision; and representativeness, completeness, and comparability of data.

2.5.3.1 Sensitivity

Sensitivity is based on the minimum concentration that a substance can be measured and reported with 99% confidence that the concentration is greater than zero. This is generally expressed in the form of the method detection limit (MDL) or quantitation limit for the analytical method selected. The equation used to calculate MDL is presented in Section 3.5.

The lowest concentration that can be reliably achieved within the specified limits of precision and accuracy during routine laboratory operating conditions is termed estimated quantitation limit (EQL). The EQL is generally 3 to 5 times greater than the MDL. The sample quantitation limit (SQL) is the quantity based on sample dilution where the EQL is multiplied by a dilution factor. If the SQL is higher than the EQL for any analysis resulting from causes other than high analyte concentrations, the project manager will discuss corrective actions with the laboratory manager and quality control officer.

Each project-specific QAPP or work plan/SAP with a site-specific QAPP Addendum will provide the concentrations of concern for contaminants known or suspected to be present at the sampling location. This information should be provided in Section 2.3 - Problem Definition and Background. The concentrations of concern will be based on risk-based criteria, regulatory limits, and other similar guidelines. The project-specific QAPP or the work plan/SAP document with site-specific QAPP Addendum will also provide the required detection limits and quantitation limits for these analytes in various matrices based upon their concentrations of concern. Quantitation limits reflect the influences of the sample matrix on method sensitivity and are typically higher than detection limits. Quantitation limits provide a more reliable indication of the amount of material needed to produce an instrument response that can be routinely identified and reliably quantified when applying a particular analytical method to real environmental samples.

For all work conducted under the EPA Environmental Services contract, the Contractor will select analytical methods with sensitivities appropriate to the intended data use. Whenever possible, analytical methods will be specified such that matrix-specific reporting limits are lower than any contaminant concentrations of concern.

2.5.3.2 Precision

Precision is a measure of the variability of a measurement system. Precision is typically estimated by means of duplicate and replicate measurements and is expressed in terms of relative percent difference (RPD). Equations for calculating RPD are presented in Section 3.5 of this Generic QAPP. For field sampling, precision is increased by following SOPs and by collecting all samples using the same sampling procedures. Field QC samples that are collected to measure precision include field duplicate samples (i.e., transport and field handling bias) and include collocated samples (i.e., sampling and measurement precision). Field measurement precision is monitored by taking replicate measurements. Field measurement precision is increased through proper operation and maintenance of field equipment.

The specific QA objectives for precision should be provided in the site-specific QAPP Addendum or Field Sampling Plan for the use of XRF equipment in analyzing soil samples.

Precision for laboratory analyses will be measured by collecting and analyzing the following types of samples: field split samples, MS/MSD samples for organic and inorganic analyses, matrix duplicate samples for inorganic analyses, and laboratory control samples (LCS) and LCS duplicate samples.

Because field and laboratory measurements and sample matrices will vary with each investigation, the specific QA objectives for precision and accuracy will be provided in the project-specific QAPP or site-specific work plan containing SAP-level QA/QC information and a completed R7 QAPP Addendum Form for SA, ISA and RSE activities. This information is presented most clearly in a table or a series of tables.

Precision is evaluated using the RPD between the results of the MS and the MSD samples. This precision evaluation can also be performed using the RPD between a blank spike (BS) and blank spikes duplicate (BSD). The spiked samples are laboratory samples that have been fortified. Precision for the fieldwork is evaluated by using the RPD between the results for the field duplicate samples. A RPD goal of +/-25% (i.e. 75% to 125%) will be used for both field and lab analyses and will be included in the task assignment. Precision determined using RPD would be calculated as follows:

$$RPD = \left[\frac{2x(X_1 - X_2)}{(X_1 + X_2)} \right] \times 100$$

where: X_1 = analyze concentration in the sample

X_2 = analyze concentration in the duplicate

2.5.3.3 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy is typically expressed as percent recovery (%R) from spiked samples or bias with respect to a reference standard. The use of spiked samples permits a constant check on method accuracy and provides an indication of the degree of matrix effect. Equations to calculate accuracy in terms of %R are presented in Section 3.5 of this Generic QAPP.

The specific QA objectives for accuracy should be provided in the site-specific QAPP Addendum or Field Sampling Plan for the use of XRF equipment in analyzing soil samples.

Accuracy for field sampling will be increased by establishing a sound sampling strategy and following appropriate SOPs. The field QC samples are collected to measure accuracy include trip blanks, field blanks, and equipment rinsate blanks. In general, the accuracy of field measurements will be increased by following appropriate SOPs and through proper calibration and maintenance of equipment. QC measures used to monitor the accuracy of field measurements include checking instrument responses against calibration standards.

Accuracy for laboratory analyses will be assessed by collecting and analyzing the following types of QC samples: MS/MSD samples for organic analyses, MS/MSD and matrix duplicate samples for inorganic analyses, and laboratory QC check samples. Additional volumes for MS/MSD samples and matrix duplicate samples are collected in the field. Other QC check samples used to assess accuracy are prepared in the laboratory. These laboratory check samples may include blank spikes, surrogate spikes, method blanks, reagent blanks, instrument blanks, calibration blanks, laboratory control samples, standard reference materials, and independent check standards.

Accuracy is evaluated by using the %R of the MS/MSDs and laboratory blank spike samples (BS/BSDs). An accuracy goal of +/- 20% of recovery (i.e. 80% to 120%) will be used. Accuracy as determined by the %R would be calculated as follows:

$$\%R = \left[\frac{X_4 - X_u}{K} \right] \times 100$$

where: X_4 = measured value of spiked sample or blank

X_U = measured value of unspoiled sample or blank

K = known amount of the spike in the sample or blank

2.5.3.4 Representativeness

Representativeness refers to the extent that the sample data precisely and accurately represent the characteristics of a group of samples, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that depends on the proper design of the sampling program and proper laboratory protocol. The sampling network for each investigation will be designed to provide data representative of environmental conditions. During development of the sampling network, consideration will be given to past waste disposal practices, existing analytical data, current and former on-site physical setting and processes, and other relevant information. This QA parameter is a measure of the design of the sampling program and use of appropriate sampling techniques, and is evaluated using the field duplicates, trip blanks, method blanks and laboratory confirmation results.

Field duplicates provide a measure of assurance that the samples are representative of the sampling point. The effects of shipping and transporting the VOC samples are assessed with trip and field blanks. Method blanks are used to determine if cross contamination has taken place in the laboratory.

Representativeness can also be affected by the time, place, and manner by which the samples are collected. In many cases, project planners account for the difficulty in knowing when, where, and how to collect representative samples by developing statistical or random sampling networks; collecting more samples than would otherwise be needed; collecting samples at several different phases of natural or anthropogenic cycles; sampling at different locations within the project area; collecting composite samples as opposed to grab samples; and verifying and validating the sampling techniques in separate studies. The site-specific study will identify specific methods for achieving and demonstrating the representativeness of the samples to be collected.

Representativeness will also be satisfied by ensuring that this Generic QAPP with appropriate site-specific QAPP Addendum is followed, samples are collected in accordance with appropriate SOPs or by proper sampling techniques when SOPs are not available, proper analytical procedures are followed, and holding times of the samples are not exceeded in the laboratory.

2.5.3.5 Comparability

This QA parameter is qualitative in signifying the confidence with which one data set can be compared with another. The sample data should be comparable to other measurement data for similar samples and sampling conditions. This parameter is achieved through standard sample collection techniques, analyses, and reporting the analytical results in appropriate units.

Generally, comparability will be attained by achieving the QA objectives for sensitivity, accuracy, precision, completeness, and representativeness given in this Generic QAPP or in the site-specific QAPP Addendum or project-specific QAPP. Following field and laboratory procedures consistently for individual investigations and for this contract will also achieve comparability of data. EPA-approved standard field procedures such as those discussed in Section 3.2 of this Generic QAPP will be used to the extent possible. EPA-approved laboratory methods such as those listed in the Contract Laboratory Program Statements of Work and in SW-846 will be used to increase the comparability of laboratory analytical data generated under this contract.

2.5.3.6 Completeness

Completeness is a measure of sample collection usability and whether the data quality has been met. Completeness is a measure of the amount of valid data obtained from a measurement system compared to the total number of measurements necessary to achieve a specified level of confidence in decision-

making. Completeness of sample collection is the ratio of the samples actually collected to the number of samples planned to be collected. The typical goal for most sample collection events is 95%. The completeness of usable data is the ratio of data that is not rejected to the total number of data points. The completeness of quality data is the ratio of data that is qualified to the total number of data points. The goals for these components are 95% and 80%, respectively. The EPA Project Manager, in consultation with the Superfund Branch Chief, will determine if the completeness goals have been met for the field and lab data. If changes to the site-specific QAPP Addendum or project-specific QAPP are necessary based on site-specific conditions, these will be documented in a QAPP revision for review and approval.

Following completion of analytical testing, the percent completeness will be calculated according to the equation presented in Section 3.5 of this Generic QAPP. In a site-specific QAPP Addendum or project-specific QAPP (where necessary) the QA objectives for completeness will be documented and explained in the site-specific QAPP Addendum or project-specific QAPP. For those sites that will include parameters other those specific to this Generic QAPP, the companion Generic QAPP (i.e., Generic Quality Assurance Project Plan for the Superfund Integrated Site Assessment and Targeted Brownfields Assessment Programs, dated July 2007) shall be used to complete the site-specific QAPP Addendum or project-specific QAPP.

2.6 Site Assessment, Integrated Site Assessment, Removal Site Evaluation and Remedial Investigations Data Categories

The data quality objectives (DQOs) for the activities performed under the SA, ISA, RSE, and RI activities should ensure that environmental data obtained meet the needs of the study and can be used with confidence to support specific decisions (both administrative and regulatory) pertaining to the site. DQOs specify the quality of data required from a particular activity to support specific decisions. Specific DQOs from the list of those outlined under this Generic QAPP will be identified and documented in the R7 QAPP Addendum Form for SA, ISA, and RSE activities, in accordance with Guidance on Systematic Planning using the Data Quality Objectives Process EPA QA/G-4 (EPA, 2006a).

There are eight removal factors listed in the NCP [NCP '300.415(b)(2)] evaluated to define the problem and therefore determine the action to be taken:

Actual or potential exposure of nearby human populations, animals, or the food chain;
Actual or potential contamination of drinking water supplies or sensitive ecosystems;
Existence of hazardous substances in containers that pose a threat of release;
Existence of highly contaminated surface soils that could migrate;
Weather conditions that could cause hazardous substances to be released or migrate;
Threat of fire or explosion;
Availability of other response or enforcement mechanisms; or
Other situations or factors that may pose a threat.

For ISA, different types (and typically greater quantities) of data are necessary than are required for a removal site evaluation alone. For example, in a site inspection and removal site evaluation, a background sample must be taken for each matrix to be analyzed, a trip blank should be used, and composite sampling gives more information economically. Additional attention is paid to pathways and possible receptors, since scoring for a site is accomplished with this data. A concerted effort is made to connect the contamination with the site sources.

2.6.1 Superfund Data Categories

Two Superfund data categories have been established, which are referred to as: 1) Screening data with or without definitive confirmation results; and 2) Definitive data. These categories segregate environmentally related measurement data into two groups, which are based primarily on increasing levels of confidence in the precision and accuracy of the analytical results. Screening data without definitive confirmation results are considered to be data of unknown quality and are preliminary in nature. Screening data with definitive confirmation results comprise data of known quality that are quantitatively "verified" and for which the analyte identification is "definitively" confirmed. Definitive data include all measurements that are performed through analyte-specific EPA-approved methodologies that definitively identify and quantify the analyte of interest. The data categories are described in more detail in Sections 3.1 and 3.2. Screening results will be used to select the type and location of analytical samples, including those that would be used for definitive confirmation. The data quality available from current field screening technologies is acceptable for this purpose.

For SA, ISA, RSE, and RI activities, either of the data categories may be used to determine the necessity for further action at the site. The appropriate category that will be used will be determined by the EPA Project Manager and will be dependent on the specific activity and required data use.

2.6.2 Screening Data With/Without Definitive Confirmation Results

The screening category is a broad classification that includes measurements that can be non-quantitative to semi-quantitative, or involve only probable identification of a compound class. This category will be appropriate for data collection activities that involve rapid, non-rigorous measurement or analytical procedures and limited quality assurance/quality control (QA/QC) requirements. The screening methods will be used to make quick assessments of the types and levels of pollutants. Screening will often be employed during SA, ISA and RSE activities and may be performed during preliminary site characterization and/or delineation of the extent of contamination across the site. The use of screening techniques will generally be confined to sites where the types of contamination are either known or suspected and/or where additional data are needed to expand on existing information.

Definitive confirmation refers to the analysis of samples by a technique that can unequivocally detect the specific analyte in question and that can produce verifiable documentation that the analyte identification is correct. A parameter of interest is considered to be valid if the precision and accuracy of the data have been determined to be within EPA-established control limits. For ISA and RSE activities, the screening data with definitive confirmation category will be applied to assessment and characterization activities where a higher level of confidence is required for the data. A minimum of 10% of the screening measurements performed under this category will be confirmed with data that meet definitive requirements. Consequently, if the results of the confirmation analyses substantiate those screening data, a higher level of confidence may be given to the remaining 90% of the screening data.

2.6.3 Definitive Data

The most exhaustive category is definitive data, which is appropriate when rigorous, EPA-approved methods of analysis and comprehensive QA/QC procedures are necessary. For ISA, RSE, and RI activities, this category will be applied when a highly significant cost or risk is associated with an incorrect decision. Definitive data are analyte-specific, with confirmation of analyte identities and concentrations. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined. See Section 2.5 for quantitative Data Quality Objectives.

2.7 Special Training Requirements or Certification

The primary training requirements for contractor personnel engaged in field activities are the emergency response and hazardous waste operations training requirements defined in 29 CFR 1910.120. However, specialized training or certification related to environmental data collection might be required if (1) specifically called for in a TO or PR, and (2) identified as necessary by contractor in responding to a TO or PR. In these situations, contractor will address training and certification needs in the site-specific QAPP Addendum or project-specific QAPP. The site-specific QAPP Addendum or project-specific QAPP will identify contractor personnel that meet the special training or certification requirements; provide documentation of the training or certification; and describe how these personnel will be assigned to the project. If contractor personnel do not meet special training or certification requirements, the site-specific QAPP Addendum or project-specific QAPP will briefly describe how the necessary skills will be acquired and applied to the project.

2.8 Documentation and Records

This section describes the requirements for data reporting that are expected of contractor field personnel and laboratories that submit field and laboratory measurement data under this EPA Environmental Services contract. It is the responsibility of the Regional Quality Assurance Manager to ensure that the latest version of the approved Generic QAPP is used. Requirements for data validation reports, data quality assessment reports, or other QC reports that are prepared or compiled by Contractor are not covered here but are described in Sections 4.1, 4.2, 5.1, and 5.2.

Each work plan with SAP and/or and site-specific QAPP Addendum or project-specific QAPP will provide the data reporting requirements for each physical or chemical field and laboratory method that is conducted during the investigation. Data reporting requirements for each field and laboratory method will depend on the DQOs and on the intended uses of the resulting data (see Sections 2.4 and 5.3). Reporting requirements must be clearly specified as part of any request for analytical services (see Section 3.4) and are closely linked to data validation requirements (see Sections 5.1 and 5.2). For example, for most inorganic analytical methods, and for metals in particular, no adequate degree of data validation can be performed without the raw data. Each work plan/SAP with site-specific QAPP Addendum or project-specific QAPP will clearly specify the data that must be reported such that (1) data validation requirements can be satisfied, and (2) attainment of DQOs can be verified.

2.8.1 Laboratory Documentation

The types of data deliverables that are often required for data produced by analytical methods include the following:

- A case narrative, including a statement of samples received, a description of any deviations from the specified analytical method, explanations of data qualifiers applied to the data, and any other significant problems encountered during analysis. The narrative will describe all QC nonconformance experienced during sample analysis, along with the corrective actions taken.
- A table that cross-references field and laboratory sample numbers.
- The chain-of-custody forms pertaining to each sample delivery group or sample batch analyzed.
- A laboratory report showing traceability to the sample analyzed and containing the following information: project identification; field sample number; laboratory sample number; sample matrix description; dates and times of sample collection, receipt at the laboratory, sample preparation, and analysis; analytical method description and reference citation; individual parameter results with concentration units (including second column results or second detector results, or other confirmatory results, where appropriate); quantitation limits

achieved; and dilution or concentration factors.

- The data summary forms and QC summary forms for sample results, surrogate results, blank results, field QC sample results, MS/MSD results, MS results, initial and continuing calibration results, confirmatory results, LCS/LCSD results, and other QC sample results.
- The laboratory control charts.
- The method detection limit and instrument detection limit results.
- The Staged Electronic Data Deliverable (SEDD) is an inter-agency effort to create a generic format for electronic delivery of analytical data for environmental programs. The data deliverable generated by SEDD is an industry-standard Extensible Markup Language (XML) file. A major advantage for laboratories is that SEDD can be implemented in stages. This allows laboratories to meet Electronic Data Deliverable (EDD) requirements for multiple programs without having to overhaul their EDD-producing systems as agency or program needs change. The requirements in http://www.epa.gov/superfund/programs/clp/sedd.htm are for EPA to CLP labs for SEDD / EDD.

Additional data deliverables may also be required depending on site-specific DQOs or on the particular field or laboratory method of concern.

Contractor Project Managers, in conjunction with the contractor's Quality Assurance Manager, have the primary responsibility for defining project-specific data reporting requirements. These requirements, the turnaround time for receipt of the data deliverables specified, and any project-specific requirements for retention of samples and laboratory records, should be clearly defined in requests for analytical services (see Section 3.4). Subcontractor's laboratory Quality Assurance Managers are responsible for ensuring that all laboratory data reporting requirements in the work plan/SAP, site-specific QAPP Addendum or project-specific QAPP are met.

The contractor will retain all project documents for a time period specified by EPA in the contract or until EPA requests transfer or disposition of the documents.

2.8.2 Field Log Books and Photographic Documentation

A field logbook (prepared by the contractor's Project Manager, subcontractor's Project Manager, or Field Team Leader) will be maintained to record all pertinent activities associated with the sampling event. Entries into the logbook will, at a minimum, be made on a daily basis. The observations and data will be recorded with waterproof ink and kept in a bound, weatherproof field logbook with consecutively numbered pages. Specific sampling information will be recorded on Field Sampling Data Sheets (an example is shown in Appendix B). Each entry into the field logbook will record the following information:

- Names of personnel present during sampling activities.
- Date, time and weather conditions.
- Name, address, and telephone number of the property owner, including private well and municipal well owners/agents.
- Equipment calibration.
- Number, types, location, sampling depth, well depth and screened interval, if available, of the wells sampled.
- Analyses performed in the field and in fixed laboratories.
- QA/QC samples collected.
- Photo log with the number (according to the roll and frame count) or file name if digital camera is used, time and a detailed description of each photo taken to record site conditions

during the sampling event.

Changes or deletions in the field logbook or sample collection field sheets will be lined out with a single strike mark and remain legible. Sufficient information will be recorded to allow the sampling event to be reconstructed without relying on the collector's memory. Each day, the person making entries in the field logbook, will sign each page with recorded information, at the end of the day. Anyone making entries in another person's field book will sign and date those entries.

Daily quality control reports (DQCRs) will be completed for each day of sampling activity by the contractor or subcontractor to supplement the information recorded in the field logbook. The DQCRs will be signed and dated by individuals making entries. A copy of the respective daily calibration logbook pages(s) will be attached to each day's DQCR. An example of DQCR is included in Appendix-C. The DQCR will be provided to the EPA Project Manager and included in site assessment reports.

2.8.3 Chain-of-Custody Documentation

All samples collected for shipment to the fixed laboratory during the study will be tracked from the time the samples are collected until laboratory data are issued. Information on the custody, handling, transfer, and transport of samples to the off-site laboratory will be recorded on a chain-of-custody (COC) form as shown in Appendix D. The sampler will be responsible for filling out the COC form. The sampler will sign the COC when relinquishing the samples to anyone else.

A COC form will be completed daily for each set of samples collected, and will contain the following information:

- Sampler's signature and affiliation
- Project name
- Sample identification numbers
- Date and time of collection
- Sample type
- Analyses requested
- Number, size and type of containers
- Preservation method
- Signature of persons relinquishing custody, including date, and time
- Signature of persons accepting custody, including date and time
- Method of shipment

The above elements are included in the latest version of EPA SOP Nos. 2420.4 "Field Chain of Custody for Environmental Samples" and 2420.5 "Identification, Documentation and Tracking of Samples". Laboratory QA/QC records and sample results will be included in the required site assessment report, perhaps as within an appendix. All documents will be kept in the EPA individual site files (hard copy) and available in the public file. All public record files are subject to the EPA Records Retention Plan as outlined in the EPA Quality Management Plan (QMP).

The site-specific task assignments and/or discussions during scope-of-work/sampling strategy meetings will allow EPA to specify the format and content for the data package as well as the desired reporting format.

3.0 DATA GENERATION AND ACQUISITION

This section of the Generic QAPP includes the 10 QAPP elements required by EPA QA/R-5 (EPA, 2000a) to address all aspects of data generation and acquisition. These QAPP elements ensure that appropriate methods for sampling, analysis, measurement and analysis, data collection or generation, data handling, and QC are identified and followed. The 10 QAPP elements related to measurement and data acquisition are:

- Sampling process design (Section 3.1)
- Sampling methods requirements (Section 3.2)
- Sample handling and custody requirements (Section 3.3)
- Analytical methods requirements (Section 3.4)
- Quality control requirements (Section 3.5)
- Inspection and equipment testing, inspection, and maintenance requirements (Section 3.6)
- Instrument and equipment calibration and frequency (Section 3.7)
- Inspection and acceptance requirements for supplies and consumables (Section 3.8)
- Non-direct measurements (data acquisition) requirements (Section 3.9)
- Data management requirements (Section 3.10)

3.1 Sampling Process Design

Sampling activities for each project will be outlined in work plans with SAP information and site-specific QAPP Addendum or project-specific QAPP. These QA/QC documents will summarize the sample network design and rationale, including: the numbers and types of samples to be collected, sampling locations, sampling frequencies, sample matrices, and measurement parameters. Key factors to be evaluated in the sampling process design include:

- Project objectives and decisions to be made.
- Information needed for the decisions and how the information will be used.
- Time and resource constraints.
- Statistical validity and legal defensibility of the data.

Completing this evaluation (1) helps ensure that the analytical results obtained fully support the decisions to be made by data users and (2) maximizes the probability of making a correct decision based on the results.

The sampling network design and rationale will be coordinated with the DQO process described in Section 2.5 of this Generic QAPP. The ultimate use of the data, as defined by the DQO process, will help determine whether grab or composite samples should be collected or whether a probability-based (statistical) data collection design or a nonrandom (judgmental) data collection design should be used.

This section also distinguishes between screening data used for information purposes only (non-critical measurements) and definitive data used to meet project objectives (critical measurements). If field-screening techniques will be used to identify samples for confirmative laboratory analysis, the site-/project-specific QA/QC documents will indicate what techniques will be used and the frequency of confirmative sampling.

For completion of this element, the site-/project-specific QA/QC documents will include a schedule table showing the anticipated start and completion dates of all major milestones, including field sampling events, laboratory analyses, data validation, and report preparation and submittal.

A generic sampling scheme for a RSE indicating an estimate of sample type, location and number of samples is shown in Table 3.1. The actual site-specific number, location and type of samples will be described in the project work plan that must include elements of a SAP and a completed R7 QAPP Addendum Form or project-specific QAPP.

Sampling will follow a biased design in that sampling locations will be from areas deemed most likely to be contaminated. Physical features such as buildings, fences, utilities, roads, lagoons, ponds, surface impoundments, etc., and access to property also will influence selection of sampling locations (based on site reconnaissance prior to sampling).

Table 3.1 – Sample Summary							
No. of Samples	**Matrix**	**Location**	**Purpose**	**Depth or other Descriptor**	**Requested Analysis**	**Sampling Methods**	**Analytical Method**
120	Soil	Residential yards, school yards, parks daycare centers	to confirm XRF readings obtained in the field	0-1 inches	arsenic, barium, cadmium, lead (total)	EPA SOPs 4231.1707 & 4231.2012	EPA Method 3050B/6010B
4	Soil/ Sediment	Stream beds, creeks, ponds, rivers, lagoons, drainage pathways	to determine whether a release to sediments has occurred	0-1 inches	arsenic, barium, cadmium, lead,	EPA SOP 4230.08A	EPA Method 3050B/6010B
2	Soil/Rock	Borrow sources, rock quarries	to determine whether possible borrow source soils and rock are non-contaminated	0-1 inches	arsenic, barium, cadmium, lead, TCLP-arsenic, barium, cadmium, lead	EPA SOPs 4231.2012 & 4231.2017	EPA Method 3050B/1311/6010B
120	Water	Residential wells in the study area	to determine whether a release to drinking water supplies has occurred	N/A	arsenic, barium, cadmium, lead (total and dissolved)	EPA SOP 4230.10A	EPA Method 6020
4	Water	Streams, creeks, ponds, rivers, lagoons, drainage pathways	to confirm whether a release to surface water has occurred	N/A	arsenic, barium, cadmium, lead (total)	EPA SOPs 4230.17A	EPA Method 6020
2	Dust/Wipe	Interior of residences	to determine whether a release within home interiors has occurred	N/A	arsenic, barium, cadmium, lead (total)	EPA SOPs 4231.2011	EPA Method 6020
2	Air	In areas potentially impacted by excavation of contaminated soils, and downwind of site repository	To confirm whether a release to the air pathway has occurred	N/A	lead (total)	NIOSH Method 7300	EPA Method 6010B
QC Samples							
12	Soil	field duplicates	to assess the precision of analytical and sampling methods	0-2 inches	arsenic, barium, cadmium, lead (total)	EPA SOPs 4231.1707 & 4231.2012	EPA Method 3050B/6010B
3	Water	field blanks	to assess field-introduced and lab-introduced contamination	N/A	arsenic, barium, cadmium, lead (total)	N/A	EPA Method 6020
10	Water	field duplicates (residential wells)	to assess the precision of analytical and sampling methods	N/A	arsenic, barium, cadmium, lead (total and dissolved)	EPA SOP 4230.10A	EPA Method 6020

3.1.1 Background Samples

Background samples may be required to compare site conditions to regional or upgradient conditions. Background samples are environmental media samples and not considered "QC samples." Criteria for utilizing background samples vary between regulatory programs. Therefore, background sampling requirements will be determined on a case-by-case basis and specified in the work plan/SAP documents, the site-specific QAPP Addendum or project-specific QAPP.

3.1.2 Site Security

The contractor will provide security at the site to protect the public and the work effort. The security level shall be sufficient to reasonably protect personal property and persons from harm or damage.

3.1.3 Disposal of Contaminated Materials

Investigation-derived waste (IDW) may consist of decontamination fluids, drill cuttings, purge/development water, excess sampled media (e.g., soil, sediment, water, etc.), disposable sampling supplies, and personal protective equipment (e.g., Tyvek/Saranex coveralls, gloves, booties, etc.). Handling of IDW will be performed according to procedures described in Management of Investigation-Derived Wastes During Site Inspections EPA/540/G-91/009 (EPA, 1991a). Attempts will be made to achieve the following goals pertaining to IDW management:

- Leave the site in no worse condition than it existed prior to site activity.
- Remove wastes that pose an immediate threat to human health or the environment.
- Leave wastes on site that do not require off-site disposal or extended containerization.
- Comply with state and federal requirements.
- Minimize the quantity of wastes generated.

Waste disposal for IDW will be dependent upon classification of the waste as either RCRA hazardous or RCRA nonhazardous.

Decontamination of personnel and equipment will be conducted in accordance with the site-specific health and safety plan and EPA Region VII guidelines.

3.1.4 Site Restoration

The contractor will repair or replace material damaged during site assessment activities and restore as near as possible the damaged environment to pre-assessment conditions. At a minimum, the contractor will perform the following:

- Regrading of surface.
- Replacement of soil.
- Replacement of damaged concrete, asphalt or other surface cover.
- Reseed or replant vegetation.

3.2 Sampling Methods Requirements

This Generic QAPP for Lead-Contaminated Sites was prepared to specifically address Superfund investigations on former and active mining, milling and smelter facilities and the associated impacted areas from the operations of these facilities. Sampling procedures may vary with each project and will be specified in the site-/project-specific QA/QC documents. This section presents information concerning the selection of sampling methods; project-specific sampling method requirements; and requirements for

containers, volumes, preservation methods, and holding times for samples that might be commonly required under the contract. Requirements for collecting QC samples are discussed in Section 3.5.

3.2.1 Sampling Methods

Sampling methods and equipment will be selected to meet project objectives. Affected media may include groundwater, surface water, sediments, surface and subsurface soils, wastes, process materials, and air. Field parameters (such as pH, specific conductance, oxidation-reduction potential, temperature, dissolved oxygen content, meteorological parameters, and water elevation) may also be measured to assist in carrying out sampling procedures effectively.

To the extent possible, the Contractor will rely on EPA-approved methods for sample collection and field measurements. EPA-approved sampling methods that are selected for use will be referenced in the site-/project-specific QA/QC documents. Guidance documents containing EPA-approved sampling SOPs include the following:

- OSWER Publication 9360.4-02. January 1991. Compendium of ERT Soil Sampling and Surface Geophysics Procedures. EPA/540/P-91/006. Interim Final. 1991b.
- OSWER Publication 9360.4-03. January 1991. Compendium of ERT Surface Water and Sediment Sampling Procedures. EPA/540/P-91/005. 1991c
- OSWER Publication 9360.4-05. May 1992. Compendium of ERT Air Sampling Procedures. PB92-963406. 1992a.
- OSWER Publication 9360.4-06. January 1991. Compendium of ERT Ground Water Sampling Procedures. EPA/540/P-91/007. 1991d.
- OSWER Publication 9360.4-07. January 1991. Compendium of ERT Waste Sampling Procedures. EPA/540/P-91/008. 1991e.
- OSWER Directive 9360.4-10. December 1995. Superfund Program Representative Sampling Guidance Volume 1: Soil. EPA/540-R-95/141. 1995a.
- OSWER Directive 9360.4-04. May 1992. Compendium of ERT Field Analytical Procedures. 1992b.
- OSWER Publication 9285.7-50. August 2003. Superfund Lead-Contaminated Residential Sites Handbook. 2003.
- Environmental Protection Agency. Pollution Prevention and Toxics. March 1995. Residential Sampling for Lead: Protocols for Dust and Soil Sampling. EPA 747-R-95-001. 1995b.
- Ground Water Sample Collection. EPA Region VII. SOP No. 4230.15.
- Surface Water Sample Collection. EPA Region VII. SOP No. 4230.17.
- Sediment Sample Collection. EPA Region VII. SOP No. 4230.08.
- Drinking Water Sample Collection. EPA Region VII. SOP No. 4230.10.
- Geoprobe Operation. EPA Region VII. SOP No. 4230.07.
- Portable XRF Analyzer, EPA Region VII. SOP No. 4231.1707.
- Chip, Wipe, and Sweep Sampling. EPA Region VII. SOP No. 4231.2011.
- Soil Sampling at Lead-Contaminated Residential Sites. EPA Region VII. SOP No. 4230.19.
- Interior Dust Sampling at Lead-Contaminated Residential Sites. EPA Region VII. SOP No. 4230.18.
- Waste Pile Sampling. EPA Region VII. SOP 4231.2017.
- X-MET™ 880 Field Portable x-Ray Fluorscence Operating Procedure. EPA Region VII. SOP No. 4232.1707.
- Spectrace 9000 Field Portable X-Ray Fluorscence Operation Procedure. EPA Region VII. SOP No. 4232.1713.

- Surface Water Sampling. EPA Region VII. SOP No. 4232.2013.
- Sediment Sampling. EPA Region VII. SOP No. 4232.2016.
- Protocols for the Region 7 Lead-Contaminated Residential Yard Soil Cleanup Actions Procedures and Sequencing. EPA Region VII. SOP No. 4220.03.

In addition, sampling methods referenced in the Region 7 ESDOQAM will be used. If an EPA-approved sampling method is not available, or a non-standard sampling method is required, the project-specific QAPP or the work plan/SAP and/or site-specific QAPP Addendum will include a procedure for the method.

Collection of groundwater samples from public and private water supply wells will follow the latest version of EPA Region 7 SOP No. 4230.10A: "Drinking Water Sample Collection", or equivalent SOPs supplied by the contractor. SOPs provided by the manufacturer for the direct push sampling probe will be followed by field personnel for soil-gas, soil and groundwater sampling.

Any boreholes created by the direct push probe will be backfilled with bentonite (or equivalent) to the surface to assure that a conduit for contaminated vapors and groundwater is not created at the site. Soil samples will be collected following the latest version of EPA SOP 4231.2012: "Soil Sampling" or equivalent SOPs supplied by the contractor and in accordance with applicable State requirements.

3.2.2 Project-Specific Sampling Methods Requirements

Although this is a Generic QAPP, there are project-specific sampling method requirements that are described in the following section. However, the following items relate to all sampling methods and requirements will be identified or referenced in the site-specific QA/QC document.

- Methods used to select sample locations for all sample matrices.
- Sampling equipment for all sample matrix types and all sampling locations.
- Support facilities with capabilities commensurate with the requirements of the sampling plan.
- Decontamination procedures for all sampling equipment (including drilling equipment). At a minimum, decontamination performed between each sampling point will involve:
    - Rinse sampling equipment with a Trisodium Phosphate or equivalent soap solution.
    - Follow with a potable water rinse.
    - Additional rinse, if required, with potable water or dionized water.
- Procedures for handling and disposing of investigation-derived wastes such as well construction wastes, decontamination fluids, disposable sampling equipment, and so forth, should follow EPA guidance document for Investigation Derived Wastes.
- Procedures for providing unique sample identification numbers that will enable personnel to accurately correlate analytical results and field information with sampling locations and field monitoring stations.

The site-/project-specific QA/QC documents will also identify personnel responsible for corrective action in cases where failures in the sampling or measurement systems occur. In general, corrective actions for field sampling and measurement failures include instrument recalibration, replacement of malfunctioning measurement instruments or sampling equipment, and recollection of samples or repeating measurements.

3.2.2.1 Surface Soil Sampling in Residential Yards, Driveways, Public Areas, and Children Play Areas

Residential properties are defined in the Superfund Lead-Contaminated Residential Sites Handbook (2003) as any area with high accessibility to sensitive populations, and include properties containing single- and multi-family dwellings, apartment complexes, vacant lots in residential areas, schools, day-care centers, community centers, playgrounds, parks, green ways, and any other areas where children may be exposed to site-related contaminated media. This document defines sensitive populations as young children (those under 7 years of age, who are most vulnerable to lead poisoning) and pregnant women. Focus is put on children less than 7 years old because susceptibility of damage during the brain development that occurs through this age range. This is the age range when children are most vulnerable to adverse cognitive effects of lead. Pregnant women are included due to the effects of lead on the fetus. Other EPA guidance and local zoning regulations should also be consulted prior to determining which properties will be treated as residential.

Lead-contaminated residential sites are defined, for the purposes of this Generic QAPP, as sites where lead is the primary contaminant of concern in residential soils. Generally, lead-contaminated sites contain other metals of concern, such as cadmium and arsenic.

It is recommended in the Handbook that when sampling residential lots with a total surface area less than 5,000 square feet (a typical urban lot size), five-point composite samples should, at a minimum, be collected from the front yard and the back yard. The front and back yard composite should be equally spaced within the respective portion of the yard, and should be outside of the drip zone and away from influences of any other painted surfaces. Composites should consist of aliquots colleted from the same depth interval. For residential lots with a total surface area greater than 5,000 square feet or with a substantial side yard consult the Handbook for additional sampling strategies.

In each sampling area, a five-aliquot composite sample will be collected from the upper 0-1 inch of soil. Aliquots will be evenly dispersed throughout each sampling area, and will be selected based on the judgment of the sample team. The sample material from a sampling area should be dried, sieved with a No. 60 screen and homogenized. The EPA Technical Review Workgroup (TRW) and American Society for Testing and Materials (ASTM) have issued guidance on sieving (ASTM, 1998; EPA, 2000c). The EPA TRW guidance addresses appropriate sieve size (No. 60) and a method for predicting the concentration in the fine fraction using concentrations measured in unsieved samples. A portion of each homogenized sample from a sampling area will be screened for lead using XRF equipment.

Additional multi-aliquot surface soil samples will also be collected from any play areas, gardens, sand piles, unpaved driveways, and any other areas which may pose a unique risk to children. The number of aliquots collected from these additional areas will depend on their size, but in general the aliquot density will be similar to sampling area sampling. For locations with no residences, the center point of the property will be established and flagged. From the center point, four sampling areas wi11 extend 100 feet in each compass direction, and the aforementioned sampling protocol will be followed (that is, a five-aliquot composite sample will be collected from each sampling area).

To evaluate the accuracy of the XRF equipment, one of every ten samples that are screened with the XRF equipment will be submitted to a lab for confirmation analysis and may include all or some of the following heavy metals: aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, molybdenum, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc. At this time, XRF equipment is not set up to collect screening level data on all the metals listed above, therefore, we only use it to screen for the target metal such as lead. Lead would be the metal that you would have a confirmation analysis performed on. The site manager will decide whether the sample submitted to the laboratory will be from the same bulk sample or the XRF specimen pack. The site

manager will also decide whether the sample should be collected in a public right-of-way or a residential yard. The soil samples submitted to the USEPA laboratory will be analyzed for heavy metals, which include aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, molybdenum, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.

See Section 4.3.2 of the Handbook (EPA, 2003) for a sampling design that is based on the assumption that removal of surficial contaminated soils and placement of a cover of clean soil will be protective of human health and the environment. Use EPA Region 7 SOP 4220.03A, Protocols for the Region 7 Lead Contaminated Residential Yard Soil Cleanup Actions Procedures and Sequencing.

3.2.2.2 Sampling of Waste Piles (Mine Tailings)

Samples of tailings should be collected to effectively characterize the tailings pile. The number of locations should be at least five locations in waste piles, but this will depend on the site specific conditions. These samples will be obtained using split spoon samplers installed using DPT and will be collected from the locations where groundwater samples will be collected. Sampling methods and protocols will follow those outlined in EPA Region 7 SOP 4230.7A, Geoprobe Operation. At each location samples from the tailings, it is recommended that samples be collected at vertical intervals of 4 feet from the surface to the base of the tailings (i.e., 0 to 4 feet, 4 to 8 feet, 8 to 12 feet, etc.). A sample from each interval will be selected for analysis. Also from each of the sample locations, a 0-1 inch depth sample should be collected and analyzed for TAL metals. In addition, the 0-1 inch samples should be sieved using a 60-mesh screen. The bulk and the fines will be analyzed for lead with the XRF equipment.

3.2.2.3 Sampling of Indoor Dust

The purpose of dust sampling is to collect data that can be included in the IEUBK model (EPA, 2002c) that will be used to prepare the human health risk assessment. The data must be expressed as a dust lead concentration in order to be used in the model. The IEUBK model does not accept lead loading data. The data from the lead-based paint assessment will be used to determine whether lead based paint is present and will be compared to the EPA criterion for determining whether lead based paint is present (1 mg/cm^2).

The main objectives for collection and analysis of dust samples and analysis of the interior paint in residential homes are to determine the extent on lead contamination in dust from residential homes and determine whether the homes that are sampled for dust also contain lead-based paint. The information collected will be included in the IEUBK model that will be used to prepare the human health risk assessment.

The amount of lead in settled dust samples can be expressed as a lead loading or as a lead concentration. Lead loading is the weight of lead per area sampled and the typical units are $\mu g/ft^2$ (EPA, 1995a). Lead concentration is the weight of lead per weight of sample and is typically reported as ug/g (EPA, 1995a). Vacuum dust collection is able to generate both lead loading and lead concentration results.
In each residence, it is anticipated that three-dust samples will be collected. Since each residence will have a different floor plan and furniture arrangement, it will not be possible to predetermine the exact sample locations. The following is a list of the three general sample areas with a description of sample location criteria based on each residence's characteristics.

***Entry Way*:** A vacuum sample will be collected from the most frequently used entry way to the residence. The sample location must be at least 1 meter (3 feet) away from the door (CS_3, Inc., 1998). If there is an option between a hard floor surface and a carpeted floor surface, the hard floor surface area will be

chosen over the carpeted surface due to the potential for better sample collection on a hard floor surface. The sample will then be collected using the appropriate vacuum method for the floor type.

***Floor*:** A floor sample will be collected from the most commonly used room in the residence other than a bedroom. The selection of the sample location is based on whether or not a child or children live at the residence. **Children are defined as less than 7 years old.** If children live at the residence, the room, other than the bedroom, where the children spend the most time on the floor in the room will be chosen. If no children live at the residence, the room, other than the bedroom, where residents spend the most time will be chosen. Sample location will be based on the floor type(s) in the room. If there is a hard floor surface and a carpeted floor surface in the room, the hard floor surface will be sampled. A sample location that is not in the main walking pathway of the room, and is also large enough to accommodate the sampling requirements, will be chosen as the sample location. The sample will then be collected using the appropriate vacuum method for the floor type.

***Bedroom*:** A sample will be collected form one bedroom in the residence. The selection of the sample location is based on whether or not a child or children live at the residence. If there is a child living at the residence, their bedroom shall be selected. If there is more than one child living at the residence, -youngest child's bedroom shall be selected. If there are no children living at the residence, the bedroom where the most time is spent shall be selected. If a child's room is selected, regardless of floor type, the sample location shall be chosen based on where the child's play area is in the room or where they spend the most time on the floor in the room. If an adult bedroom is selected, the sample shall be collected based on floor type. In that bedroom, if there is a hard floor surface and a carpeted floor surface in the room, the hard floor surface will be sampled. Once the sample location has been determined, the sample will then be collected using the appropriate vacuum method for the floor type. All dust samples collected during the investigation will be sent to the EPA Region VII laboratory for analysis of TAL metals.

3.2.2.3.1 Vacuum Sampling

This method of dust sampling is suitable for the collection of settled dust samples from both hard and smooth or highly textured surfaces, such as brickwork and rough concrete, and soft, fibrous surfaces, such as upholstery and carpeting. This method produces samples for lead determination results in both loading ($\mu g/ft^2$) and concentration ($\mu g/g$).

Equipment for sampling is described in the American Society for Testing Materials (ASTM) Standard D5438-05 (ASTM, 2005). A minimum volume of dust sample may be required by the laboratory to analyze the sample for metals. This minimum volume of dust sample should be included in the site specific QAPP Addendum or project-specific QAPP.

3.2.2.3.2 Carpet Floor Sampling

Dust samples from carpeted floors will be collected in accordance with Paragraph 11.1 of ASTM D5438-05. Dimensions of the sampled area must be noted on the Field Sheet. The dust sample should be placed in a clean 4 oz wide mouth sample jar to be sieved and weighed. A 60-mesh sieve will be used to sieve the dust sample.

3.2.2.3.3 Hard Surface Floor Sampling

Dust samples from carpeted floors shall be collected in accordance with Paragraph 11.2 of ASTM D5438-05 (ASTM, 2005). Dimensions of the sampled area must be noted on the Field Sheet. The dust sample should be placed in a clean 4 oz wide mouth sample jar and taken to be sieved and weighed. A 60-mesh sieve will be used to sieve the dust sample.

3.2.2.4 Interior and Exterior Lead-Based Paint Assessment Paint

Lead-based paint (LBP) screening will be conducted on the interior and exterior of homes at properties where dust samples are collected. The purpose of the LBP screening is to determine whether LBP is present in the home. The LBP screening performed is not intended to be as comprehensive as a LBP inspection or a lead hazard screen as defined in the EPA regulation at 40 CFR 745.227.

Lead screening readings will be taken using XRF equipment. The XRF equipment will provide LBP data in milligrams per square centimeter (mg/cm^2). The unit is capable of analyzing LBP to less than 1 mg/cm^2. Forms for recording XRF readings in homes, documenting calibration checks, and recording substrate correction values, if required, are presented in ASTM D5438-05 (ASTM, 2005).

The following procedures should be followed during the LBP screening assessments:

- Conduct an initial visual inspection of the exterior walls of the home and the interior painted surfaces in rooms where dust samples are collected and assess whether significant of chipping, peeling and/or deteriorating paint is present.

- If significant deteriorating painted surfaces are observed on the exterior walls of the residence, each of the four walls of the residence will be analyzed for LBP using XRF equipment. If significant deteriorating painted surface are observed in the interior rooms where the dust samples are collected, each of the four walls in the room and a minimum of two window sills will be analyzed for LBP using XRF equipment. The XRF readings will be taken at the location of the deteriorating painted surfaces.

- If deteriorating painted surfaces are not observed on the exterior walls of the residence, each of the four walls of the residence will be analyzed for LPB using XRF equipment. If deteriorating paint is not found observed in the rooms where dust sampled are collected, XRF readings will be taken from each of the four walls and a minimum of two window sills.

- The sampling team will document the general description of the interior walls and window sills in the rooms where XRF readings are taken.

3.2.3 Sample Container, Volume, Preservation, and Holding Time Requirements

When appropriate, each project work plan with a SAP and a R7 QAPP Addendum Form or project-specific QAPP (for SA, ISA and RSE activities) will specify the required sample volume, container type, preservation technique, and holding time for each analysis to be conducted on each sample matrix. This information will most likely be presented in tabular form.

3.3 Sample Handling and Custody Requirements

Each sample collected by the contractor under this contract must be traceable from the point of collection through analysis and final disposition to ensure sample integrity. Sample integrity helps ensure the legal

defensibility of the analytical data and subsequent conclusions. The team will use standard EPA procedures to identify, track, monitor, and maintain chain-of-custody for all samples. Chain-of-custody records will establish the documentation necessary to trace sample possession from collection through final disposition. Each person that has custody at any time throughout the sample history is held responsible for maintaining proper documentation and control measures. A sample is under a person's custody if it:

- Is in that person's possession.
- Is in that person's view after being in his or her possession.
- Is in that person's possession and he or she places it in a secured location.
- Is placed by that person in a designated secure area.

Samples will be handled in accordance with the following national guidance documents and EPA Region VII SOPs:

- Field Chain of Custody for Environmental Samples. EPA Region VII. SOP No. 2420.04C.
- Identification, Documentation, and Tracking of Samples. EPA Region VII. SOP No. 2420.05D.
- Sample Container Selection, Preservation, and Holding Times. EPA Region VII. SOP No. 2420.06E.
- Sample Receipt & Log-In. EPA Region VII. SOP No. 2420.01E.
- RLB Procedures for Preparation of Field Sheets and Tags. EPA Region VII. SOP No. 2420.13C

Field and laboratory chain-of-custody procedures are discussed in the next section.

3.3.1 Field Chain-of-Custody Procedures

All projects conducted by the contractor under this contract will follow sample and document control procedures, sample and evidence identification procedures, field records requirements and procedures, and chain-of-custody procedures outlined in the latest version of EPA Region 7 SOPs 2420.04C and 2420.05D. Samples will be packaged and labeled for shipment in compliance with current U.S. Department of Transportation (DOT) and International Air Transport Association (IATA) dangerous goods regulations. Any additional requirements stipulated by the overnight shipping firm will be followed.

3.3.1.1 Field Procedures

The sample packaging and shipment procedures summarized below will ensure that the samples arrive at the laboratory with the chain-of-custody intact. All chain-of-custody forms should be filled out in ink. Certain information required on the chain-of-custody form such as names of samplers, and date and time of sample collection are self-explanatory. The following additional information will be entered on the chain-of-custody form:

- The Contractor task order number will be entered in the space entitled "project number."
- A description of where the sample was taken will be included in the space entitled "station location" (for example, southwest corner of drum storage area).

- The target parameters and analytical method will be entered in the space entitled "analysis required" (for example, metals, SW846 Method 6010B).

The contractor will use EPA Region 7 SOP No. 2420.06E, "Sample Container Selection, Preservation, and Holding Times", or equivalent SOPs. The contractor field personnel will follow the steps outlined below to prepare the samples and custody documents:

- Immediately after sample collection, sample containers will be labeled with the appropriate identifiers, and clear tape will be placed over the labels to preserve label integrity.
- The samples will be placed in Ziploc™ plastic bags and which will then be immediately placed on ice in cooler containing double-sealed bags of ice and maintained at 4° C.
- Glass containers will be wrapped in bubble pack and placed in Ziploc™ plastic bags. Samples will be transported or shipped to the laboratory in time so that the analysis can be performed before the holding times are exceeded.
- Prior to shipping, the chain-of-custody forms, airbills, and all other relevant documents will be completed. Chain-of-custody forms will be sealed in plastic bags and taped to the inside of the cooler lid. Cushioning material, consisting of bubble-wrap, will be placed in the cooler.
- A temperature blank consisting of a jar or vial containing water will be included in every cooler to be used by the laboratory to determine the cooler temperature at the time of sample receipt.
- The shipping cooler will then be sealed with tape and custody seals in a manner that will indicate whether the cooler was opened. The preferred procedure includes placement of custody seals at diagonally opposite corners of the cooler. The custody seals will be covered with clear plastic tape or strapping tape.
- Coolers will remain in a secured area or in view of the sampler until it is properly sealed for shipment to the laboratory.

During field sampling activities, traceability of the sample must be maintained from the time the samples are collected until laboratory data are issued. Information on the custody, transfer, handling, and shipping of samples to the off-site laboratory will be recorded on a chain-of-custody (COC) form. Details of the chain of custody requirements are discussed in Section 2.8.3.

Contractor will utilize the following the latest version of EPA Region 7 SOPs, or similar and equivalent SOPs for handling and tracking samples:

- 2420.04C, "Field Chain-of-Custody for Environmental Samples."
- 2420.05D, "Identification, Documentation and Tracking of Samples."
- 2420.006E, "Sample Container Selection, Preservation, and Holding Times."
- 2420.11D, "Preparation of Aqueous and Soil Trip Blanks."
- 2420.12C, "Preparation of Chemical Preservatives for Aqueous Environmental Samples."
- 2420.13C, "RLAB Procedures for Preparation of Field Sheets and Tags."

The field sampler is personally responsible for the care and custody of the samples until they are transferred to other Contractor personnel or properly dispatched to an overnight carrier or directly to a laboratory. As few people as possible should handle the samples to prevent loss, breakage, or potential contamination. When transferring possession of the samples, the individuals relinquishing and receiving the samples sign, date, and note the time of transfer on the chain-of-custody form. Commercial carriers are not required to sign off on the chain-of-custody form as long as the form is sealed inside the sample cooler and the custody seals remain intact.

3.3.1.2 Field Logbooks

Field logbooks provide the means of recording all data collection activities performed. Logbook entries will be described in as much detail as possible so that a particular situation can be reconstructed without relying on memory. Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel but will be stored in the secure location when not in use. Each logbook will be identified by a site-specific document number. The title page of each logbook will contain the following information:

- Person to whom the logbook is assigned.
- Logbook number.
- Project name.
- Project start and end dates.

All logbook entries will be made in ink and no erasures will be made. If an incorrect entry is made, the incorrect information will be crossed out with a single strike mark that will be initialed and dated by the person making the correction. Logbook entries will contain a variety of information. The beginning of each entry will note the date, start time, weather, name of all team members' present, facility visitors present and the purpose of their visit, level of personal protection used, and signature of the person making the entry.

Whenever a sample is collected or a measurement is taken, a detailed description of the sampling or measurement location, which may include compass and distance measurements, will be recorded in the logbook. The number and description of any photographs taken of the location will also be noted. All equipment used to take measurements will be identified along with the date of equipment calibration. The equipment used to collect samples will be noted along with the time of sampling, sample description, depth at which the sample was collected, sample volume, number of containers, and sample preservation method. The number, type, and location of QC samples will also be noted in the logbook.

3.3.2 Laboratory Chain-of-Custody Procedures

Custody procedures must be followed in the laboratory from sample receipt until the sample is discarded. The procedures required for this contract are those required by the EPA Contract Laboratory Program (CLP) Statements of Work (SOW). These procedures are described in this section.

The laboratory should designate a specific person as the sample custodian, with an alternate designated to act in the custodian's absence. The custodian will receive all incoming samples and indicate receipt by signing the accompanying custody forms and retaining copies of the signed forms as permanent records. Once the sample transfer process is complete, the laboratory is responsible for maintaining internal logbooks, lab tracking reports, and other records necessary to maintain custody throughout sample preparation and analysis.
The laboratory sample custodian will record all pertinent information concerning the sample, including the persons delivering and receiving the sample, the date and time received, the method by which the sample was transmitted to the laboratory, sample condition at the time of receipt (sealed, unsealed, or broken container; temperature; or other relevant remarks), the sample identification number, and any unique laboratory identification number associated with the sample.

The laboratory must provide a secure storage area, restricted to authorized personnel, for all samples. The custodian will ensure that samples that are heat- or light-sensitive, radioactive, have other unusual physical characteristics, or require special handling are properly stored and maintained prior to analysis. Only the custodian can distribute samples to laboratory personnel authorized to conduct the required analyses. Laboratory analytical personnel are responsible for the care and custody of the sample upon

receipt. These personnel must be prepared to testify that the sample was in their custody at all times from the moment they received it from the custodian until the time that the analyses were completed.

At the completion of sample analysis, any unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned tagged sample should be retained in secure storage until the custodian receives permission to dispose of the sample. Sample disposal will occur only on the order of the laboratory director, in consultation with EPA or contractor, or when it is certain that the information is no longer required or the samples have deteriorated. Likewise, tags and laboratory records will be maintained until the information is no longer required and final disposition is ordered by the laboratory director, in consultation with EPA or the contractor.

3.4 Analytical Methods Requirements

The source of analytical services to be provided will in part be determined by DQOs, the intended use of the resulting data, and TO or PR-specific requirements and constraints such as quick turnaround of data. The work plan and QA/QC documents or the project-specific QAPP will identify the specific laboratory that has been selected to provide analytical services.

This section of the Generic QAPP outlines the procedures that the contractor will use to identify and select field and laboratory analytical methods that are consistent with DQOs.

3.4.1 Field Analytical Methods

Whenever possible, the contractor will use EPA-approved methods for field measurements and analyses. For example, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, and SW-846 may be used to determine field parameters such as pH, specific conductance, dissolved oxygen, and temperature. For situations where an EPA-approved or standard method does not exist, or where a modification of an EPA-approved method is used, the contractor will include appropriate SOPs in the project work plan and site-specific QAPP Addendum or project-specific QAPP. The SOPs must contain method performance study information to confirm the performance of the method for each applicable matrix. If previous performance studies are not available, they must be developed during the project and included as a part of the project results.

3.4.2 Laboratory Analytical Methods

To select appropriate methods for sample preparation, cleanup, and analysis, the contractor will consider the specific parameters of interest, sample matrices, and minimum detectable concentrations needed to accomplish project DQOs. Whenever possible, the contractor will select methods from SW-846 or from the Contract Laboratory Program Statements of Work for Organics and Inorganics Analyses. If these sources do not include an analytical method consistent with DQOs, the contractor will review other EPA-approved methods such as those specified in SW-846. Table 3.2 includes a listing of some of the more commonly used analytical methods found in the SW-846 compendium of methods.

Table 3.2 EPA Approved Methods					
Matrix	**Location**	**Purpose**	**Requested Analysis**	**Sampling Methods**	**Analytical Method**
Soil	Residential yards, school yards, parks daycare centers	to confirm XRF readings obtained in the field	arsenic, barium, cadmium, lead (total)	EPA SOPs 4231.1707 & 4231.2012	EPA Method 3050B/6010B
Soil/ Sediment	Stream beds, creeks, ponds, rivers, lagoons, drainage pathways	to determine whether a release to sediments has occurred	arsenic, barium, cadmium, lead,	EPA SOP 4230.08A	EPA Method 3050B/6010B
Soil/Rock	Borrow sources, rock quarries	to determine whether possible borrow source soils and rock are non-contaminated	arsenic, barium, cadmium, lead, TCLP-arsenic, barium, cadmium, lead	EPA SOPs 4231.2012 & 4231.2017	EPA Method 3050B/1311/6010B
Water	Residential wells in the study area	to determine whether a release to drinking water supplies has occurred	arsenic, barium, cadmium, lead (total and dissolved)	EPA SOP 4230.10A	EPA Method 6020
Water	Streams, creeks, ponds, rivers, lagoons, drainage pathways	to confirm whether a release to surface water has occurred	arsenic, barium, cadmium, lead (total)	EPA SOPs 4230.17A	EPA Method 6020
Dust/Wipes	Interior of residences	to determine whether a release within home interiors has occurred	arsenic, barium, cadmium, lead (total)	EPA SOPs 4231.2011	EPA Method 6010B
Air	In areas potentially impacted by excavation of contaminated soils, and downwind of site repository	To confirm whether a release to the air pathway has occurred	lead (total)	NIOSH Method 7300	EPA Method 6010B
QC Samples					
Soil	field duplicates	to assess the precision of analytical and sampling methods	arsenic, barium, cadmium, lead (total)	EPA SOPs 4231.1707 & 4231.2012	EPA Method 3050B/6010B
Water	field blanks	to assess field-introduced and lab-introduced contamination	arsenic, barium, cadmium, lead (total)	N/A	EPA Method 6020
Water	field duplicates (residential wells)	to assess the precision of analytical and sampling methods	arsenic, barium, cadmium, lead (total and dissolved)	EPA SOP 4230.10A	EPA Method 6020

When EPA-approved methods are not available or appropriate for project-specific requirements, other recognized standard analytical methods, such as those published by the ASTM or the National Institute for Occupational Safety and Health (NIOSH), may be used.

The published methods mentioned are updated at various time intervals. Hence, both old and new versions of these published methods exist, and future updates of these published methods will also be produced. Unless otherwise stated, laboratories conducting work under the EPA Environmental Services contract will use the most current version of any specified analytical method.

The EPA Analytical Service Request (ASR) form will be used to schedule sample analyses to be conducted by the EPA Region 7 Laboratory.

Laboratory analytical methods will vary with each investigation conducted under the contract and should be identified in the site-/project-specific QA/QC documents. When laboratory analyses will be conducted exactly according to the most recent EPA-approved methods listed above, the QAPP will reference the appropriate method. However, for some EPA-approved methods, it may be necessary to include additional information in the site-/project-specific QA/QC documents. For example, some methods found in SW-846 allow the user to specify digestion methods for soil samples. The specific options selected will be included in the Analytical Service Request form and in the site-/project-specific QA/QC documents.

On rare occasions, project-specific conditions might require the use of an analytical method that is either a modification of an EPA-approved method or is not an EPA-approved or standard method. These methods will typically be provided by the laboratory performing the method and will include a detailed description of sample preparation, instrument calibration, sample analyses, method sensitivity, associated QA/QC requirements, and acceptance criteria. The laboratory or method developer must provide method performance study information to confirm the performance of the method for each applicable matrix; if previous performance studies are not available, they must be developed during the project and included as part of the project results.

If an analytical system fails, the contractor's Quality Assurance Manager will be notified and corrective action will be taken. In general, corrective actions will include stopping the analysis, examining instrument performance and sample preparation information, and determining whether instrument recalibration and re-preparation and reanalysis of samples are warranted.

3.5 Quality Control Requirements

Quality Assurance/Quality Control (QA/QC) samples will be collected to evaluate the precision and accuracy of the mobile and fixed laboratory analysis.

Various kinds of field and laboratory QC samples and measurements will be used to verify that analytical data meet project-specific QA objectives. Field QC samples and measurements will be used to assess how the sampling activities and measurements influence data quality. Similarly, laboratory QC samples will be used to assess how a laboratory's analytical program influences data quality. The work plan with site-specific QAPP Addendum or project-specific QAPP will provide a description (usually in table format) of the QC samples to be analyzed during the investigation for (1) each field and laboratory environmental measurement method and (2) each sample matrix type.

This section of the Generic QAPP provides definitions and typical collection and analysis frequencies for common field and laboratory QC samples and measurements. In addition, this section outlines the procedures used to assess field measurements, laboratory data, and common data quality indicators.

3.5.1 Field Quality Control Requirements

Field QC samples will be collected and analyzed to assess the quality of data generated from sampling activities. These samples may include trip blanks, field blanks, equipment rinsate blanks, field duplicates, field split samples, matrix spike (MS) samples, matrix spike duplicate (MSD) samples, and matrix duplicate samples. Field QC measurements may include field replicate measurements and checks of instrument responses against QC standards.

Trip blanks, field blanks, and equipment blanks should be free of contaminants. If contaminants are detected, the data from the environmental samples may be qualified as per data validation procedures discussed in Section 5.

Trip blanks are used to assess the potential for sample contamination during handling, shipment, and storage. Trip blanks will consist of VOC analysis vials filled with ASTM Type II water at the laboratory. The trip blanks are sealed and transported to the field; kept with empty sample bottles and then with investigative samples throughout the field effort; and returned to the laboratory for analysis with the investigative samples. Trip blanks are never opened in the field. One trip blank will be included within every shipping cooler of liquid samples to and from the field to be analyzed for VOCs to detect any cross-contamination during handling and transport.

Field blanks are samples of the same or similar matrix as the actual investigative samples that are exposed to the sampling environment or equipment at the time of sampling. They are used to assess

contamination resulting from ambient conditions. Field blanks are generally not required for solid matrices but may be collected on a case-by-case basis.

Equipment rinsate blanks are collected when sampling equipment is used. These blanks assess the cleanliness of sampling equipment and the effectiveness of equipment decontamination. Equipment rinsate blanks are collected by pouring analyte-free water over surfaces of cleaned sampling equipment that contact sample media. Equipment rinsate blanks are collected after sampling equipment has been decontaminated but prior to being reused for sampling. Equipment rinsate blanks are typically collected for each type of decontaminated sampling equipment.

Field duplicate samples are independent samples collected as close as possible in space and time to the original investigative sample. Immediately following collection of the original sample, the field duplicate sample is collected using the same collection method. Care should be taken to collect the field duplicate sample as close to the location of the original sample as possible. Field duplicate samples can measure how sampling and field procedures influence the precision of an environmental measurement. They can also provide information on the heterogeneity of a sampling location. One field duplicate groundwater sample per site will be collected (sequentially) at a frequency of one for every 10 investigative samples of the same matrix type. A minimum of one field duplicate sample should be taken for each matrix sampled, even if less than 10 samples are collected for the applicable matrix. Field duplicates will be analyzed at the fixed laboratory for the same parameter as the primary sample analyzed at the on-site lab. A duplicate soil sample per site will be collected from the same sampling spoon as the primary sample. These results will be used to evaluate the representativeness of the sample.

Field split samples are usually a set of two or more samples taken from a larger homogenized sample. The larger sample is usually collected from a single sampling location, but can also be a composite sample. Field split samples can be sent to two or more laboratories and are used to provide comparison data between the laboratories. Regulatory agencies involved in a project may request that field split samples be collected to monitor how closely laboratories are meeting site-/project-specific QA objectives.

MS/MSD samples are often collected for analysis by inorganic methods. Solid MS/MSDs usually require no extra volume. MS and matrix duplicate samples are typically collected for inorganic analysis. The MS sample and matrix duplicate sample are each a single sample, usually collected from a single location at double the normal sample volume. In the laboratory, MS/MSD samples and MS samples are spiked with known amounts of analytes. Matrix duplicate samples are not spiked. Analytical results of MS/MSDs are used to measure the precision and accuracy of the laboratory inorganic analytical program and MSs are used to measure the accuracy of the inorganic analytical program. Matrix duplicate sample are used to measure the precision of the inorganic analytical program. Each of these QC samples is typically collected and analyzed at a frequency of one for every 20 investigative samples per matrix. QC checks for field measurements will consist primarily of initial and continuing calibration checks of field equipment. When applicable, QC check standards independent of the calibration standards will be used to check equipment performance. For example, when checking the accuracy of field equipment such as pH meters, a standard buffer solution independent of the calibration standards may be used. Precision of field measurements will usually be checked by taking replicate measurements. To the extent possible, Contractor will use EPA-approved field methods. If approved methods are not available, Contractor SOPs will be referenced in the project work plan and/or site-/project-specific QA/QC documents. The types and frequencies of field QC measurements and the QC limits for these measurements will be specified in the project's QA/QC documents.

3.5.2 Laboratory Quality Control Requirements

The laboratory QA/QC elements including laboratory spikes and blanks will be performed in accordance with the latest versions for EPA analytical methods SOPs and EPA Region 7 SOP No. 2430.12E, "Regional Laboratory Quality Control Policy", or equivalent SOP supplied by contractor. The EPA Project Manager will be responsible for verifying that copies of the referenced SOPs are available and

that the SOPs are being followed by conducting periodic site visits to the field, mobile lab and fixed laboratory. When the contractor has identified the subcontractor for the mobile and fixed laboratory, copies of the laboratory's SOPs will be acquired and added as an addendum to the site-/project-specific QAPP.

All water samples will require fixed laboratory confirmation. In general, fixed laboratory analysis will be performed for all critical samples needed to establish primary targets, support attribution, and/or otherwise used for site scoring.

All laboratories that perform analytical work under this EPA environmental services contract must adhere to a QA program that is used to monitor and control all laboratory QC activities. Each laboratory must have a written QA manual that describes the QA program in detail. The laboratory Quality Assurance Manager is responsible for ensuring that all laboratory internal QC checks are conducted according to the laboratory's QA manual, the requirements of this Generic QAPP, and any additional requirements included within a project-specific QAPP.

Many of the laboratory QC procedures and requirements are described in EPA-approved analytical methods, laboratory method SOPs, and method guidance documents. However, if laboratory QC requirements are not specified in an analytical method, or if additional requirements beyond those included in an analytical method are necessary to ensure that project QA objectives and DQOs are met, the project-specific QAPP will identify the additional laboratory QC checks that must be performed. The following types of information should be included:

- Laboratory analytical methods to which the internal QC checks applies.
- Complete procedures for conducting the internal QC check.
- QC samples and QC measurements involved in the internal QC check.
- Complete collection and preparation procedures for the QC samples.
- Spiking analytes and concentrations.
- Control limits for the internal QC check.
- Corrective action procedures to be followed if the internal QC checks are not done properly or results are outside control limits.

Laboratory QC procedures and requirements may include the preparation and analysis of laboratory control samples (LCS), method blanks, MS and MSD samples, surrogate spikes, and standard reference materials or independent check standards. QC checks that are most frequently required are discussed in the following sections.

3.5.2.1 Laboratory Control Samples

Laboratory control samples (LCS) are well-characterized, laboratory-generated samples that will be used to monitor the laboratory's day-to-day performance of analytical methods. LCSs can include laboratory duplicate samples, laboratory spike samples, or method blanks. The results of LCS analyses are compared to well-defined laboratory control limits to determine whether the laboratory system is in control for the particular method. If the system is not in control, corrective action is implemented. Corrective action can include stopping the analysis; examining instrument performance or sample preparation and analysis information; and determining whether re-preparation or reanalysis is warranted.

3.5.2.2 Method Blanks

Method blanks, also known as analytical process or preparation blanks, are analyzed to assess the level of background interference or contamination that exists in the analytical system and that may lead to the

reporting of elevated concentration levels or false-positive or false-negative data. One method blank is typically analyzed for every 20 samples processed by the analytical system. For batches smaller than 20 samples, one method blank is analyzed with every batch of samples processed.

A method blank consists of reagents specific to the analytical method that are carried through every aspect of the analytical procedure, including sample preparation, cleanup, and analysis. Results of the method blank analysis are evaluated in conjunction with other QC information to determine the acceptability of the data generated for that batch of samples. Ideally, the concentration of target analytes in the method blank should be below the method or instrument detection limit for that analyte. For some common laboratory contaminants, detection of a higher concentration may be allowed.

If the blank for any analysis is not within control limits, the source of contamination must be investigated, and appropriate corrective action must be taken and documented. Investigation includes an evaluation of the data to determine the extent and effect of the contamination on sample results. If a method blank indicates analytes above the method or instrument detection limits, an investigation should be conducted to determine whether any corrective action could eliminate an ongoing source of target analytes.

Refer to the individual analytical methods and the appropriate data validation guidance documents for detailed information regarding blank frequencies of analyses, acceptance criteria for blanks, and corrective actions for out-of-compliance blank results (see Section 5.2).

3.5.2.3 Matrix Spikes, Matrix Spike Duplicates, and Matrix Duplicates

A matrix spike (MS) is an environmental sample to which known concentrations of target analytes have been added. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis. If the number of target analytes is large, target analytes are divided into two to three spike standard solutions. Each spike standard solution must be alternately used. The MS, in addition to an unspiked aliquot, is taken through the entire analytical procedure, and the recovery of the analytes is calculated. Results are expressed as percent recovery (%R). One MS is typically analyzed for every 20 investigative samples prepared in one batch for inorganic analyses.

An MS/MSD is an environmental sample divided into two separate aliquots, each of which is spiked with known concentrations of target analytes. The two spiked aliquots, in addition to an unspiked sample aliquot, are analyzed separately, and the results are compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as relative percent difference (RPD) and percent recovery (%R) and are compared to control limits that have been established for each analyte. If results fall outside control limits, corrective action must be performed. One MS/MSD is typically analyzed for every 20 investigative samples prepared in one batch for organic or inorganic analyses.

A matrix duplicate sample is an environmental sample divided into two separate aliquots that are analyzed separately. The results are compared to determine the effects of the matrix on analytical precision. Results are expressed as RPD and are compared to control limits established for each analyte. If results fall outside control limits, corrective action must be performed. One matrix duplicate sample is typically analyzed for every 20 investigative samples prepared in one batch for inorganic analyses.

3.5.2.4 Standard Reference Materials and Independent Check Standards

Standard reference materials and independent check standards can be used to evaluate the accuracy of an analytical system. The source, traceability, certification of purity, and concentration of these materials and standards must be documented. The “true” known concentrations of standard reference materials and independent check standards is then compared to results obtained from the analytical system to evaluate the accuracy of the system.

3.5.3 Common Data Quality Indicators

This section describes how QA objectives for precision, accuracy, completeness, and sensitivity are measured, calculated, and reported. For some investigations, additional equations might also be needed (for example, equations for calculating mass balances, emission rates, and confidence ranges).

3.5.3.1 Precision

Precision of many analyses is assessed by comparing analytical results of MS/MSD sample pairs for organic and inorganic analyses, field duplicate samples, field split samples, laboratory matrix duplicate samples, and replicate measurements. If calculated from two measurements, precision is normally measured as RPD:

$$RPD = \left[\frac{2\ x\ (C_1 - C_2)}{(C_1 + C_2)} \right] \times 100$$

where: RPD = Relative percent difference
C_1 = Larger of the two observed measurement values
C_2 = Smaller of the two observed measurement values

For field measurements such as pH, where the absolute variation is more appropriate, precision is often reported as the absolute range (D) of duplicate measurements:

$$\%D = |m_1 - m_2|$$

where: D = Absolute range
m_1 = First measurement value
m_2 = Second measurement value

3.5.3.2 Accuracy

The accuracy of many analytical methods is assessed using the results of MS/MSD samples for organic and inorganic analyses, MS samples for inorganic analyses, surrogate spike samples, laboratory control samples, standard reference materials, independent check standards, and measurements of instrument responses against zero and span gases. For measurements where spikes are used, %R is often calculated as a measure of accuracy:

$$\%R = 100 \times \left[\frac{(S - U)}{C_{sa}} \right]$$

where: $\%R$ = Percent recovery
S = Measured concentration in spiked aliquot
U = Measured concentration in unspiked aliquot (usually equals zero for surrogate spikes)
C_{sa} = Actual concentration of spike added

When a standard reference material (SRM) is used, the following equation is often used to calculate %R:

$$\%R = 100 \times \left[\frac{C_m}{C_{srm}} \right]$$

where: $\%R$ = Percent recovery
C_m = Measured concentration of SRM

C_{srm} = Actual concentration of SRM

For field measurements such as pH, accuracy is often expressed in terms of bias (B) and is calculated as follows:

$$B = M - A$$

where: M = Measured value of SRM
A = Actual value of SRM

3.5.3.3 Completeness

Completeness is defined as follows for most measurements:

$$\%C = 100 \times \left[\frac{V}{n} \right]$$

where: $\%C$ = Percent completeness
V = Actual number of measurements judged valid (the validity of a measurement result is determined by judging its suitability for its intended use)
n = Total number of measurements planned to achieve a specified level of confidence in decision making

3.5.3.4 Sensitivity

The achievement of method detection limits (MDL) depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to ensure data quality and to ensure that analyses meet the QA objectives for sensitivity stated in the project QA/QC documents. Method sensitivity is typically evaluated in terms of the MDL and is defined as follows for many measurements:

$$MDL = t(n-1,1-x=0.99)s$$

where: MDL = Method detection limit
s = Standard deviation of the replicate analyses
$t_{(n-1,\,1-\alpha=0.99)}$ = Student's t-value for a one-sided 99 percent confidence level and a standard deviation estimate with n-1 degrees of freedom
n = Number of measurements
x = Statistical significance level

3.6 Instrument and Equipment Testing, Inspection, and Maintenance Requirements

This section outlines testing, inspection, and maintenance procedures for field equipment and instruments and for laboratory instruments. This section includes general requirements applicable to both field and laboratory equipment as well as field-specific and laboratory-specific requirements.

3.6.1 General Requirements

General requirements for testing, inspection, and maintenance procedures for the Environmental Services contract are as follows. Testing, inspection, and maintenance methods and frequency will be based on the type of instrument; its stability characteristics; the required accuracy, sensitivity, and precision; it's intended use, considering project-specific DQOs; manufacturer's recommendations; and other conditions affecting measurement or operational control. For most instruments, preventive maintenance is

performed according to procedures and schedules recommended in (1) the instrument manufacturer's literature or operating manual or (2) SOPs associated with particular applications of the instrument. In such cases, the project work plan and QA/QC documents or site-/project-specific QAPPs will reference these documents for the testing, inspection, and maintenance procedures and schedules to be used. The site-specific QAPP Addendum or project-specific QAPP will also reference these documents and/or will provide in the body of the site-specific QAPP Addendum or project-specific QAPP, how the availability of critical spare parts will be assured and maintained for all instruments and applications used for the project.

In some cases, testing, inspection, and maintenance procedures and schedules may differ from the manufacturer's specifications or SOPs. This can occur when a field instrument is used to make critical measurements or when the analytical methods associated with a laboratory instrument require more frequent testing, inspection, and maintenance. In these situations, any special testing, inspection, and maintenance procedures and schedules will be outlined in the project work plan documents and/or the site-specific QAPP Addendum or project-specific QAPP.

Any field or laboratory instrument that is in disrepair or is out of calibration must be segregated, clearly marked, and not used until it is repaired and recalibrated. If an instrument is repeatedly broken or out of calibration, the instrument must be replaced or repaired so that it is in good working order. When the condition of an instrument is suspect, unscheduled testing, inspection, and maintenance must always be conducted. Adherence to these field and laboratory preventive maintenance practices is subject to verification during performance and system audits.

3.6.2 Field Equipment and Instruments

The contractor is responsible for (1) thoroughly checking and calibrating each instrument before shipment to the field and (2) including instructions for field calibration, testing, and maintenance of each instrument shipped. Once in the field, the contractor field team leaders assume responsibility for testing, inspection, and maintenance of field instruments and equipment.

Field equipment and instruments will be inspected for damage after arrival in the field. Damaged equipment and instruments will be immediately replaced or repaired. Battery-operated equipment is checked to assure full operating capacity; if needed, batteries are recharged or replaced. Critical spare parts such as tape, paper, pH probes, electrodes, batteries, and battery chargers will be kept on site to minimize equipment downtime. Backup instruments, equipment, and additional spare parts will be available on site or within a 1-day shipping period to avoid delays in the field schedule.

Following use, field equipment will be properly decontaminated prior to being returned to its source. When the equipment is returned, copies of any field notes regarding equipment problems will be included so that problems are not overlooked and any necessary equipment repairs are carried out.

3.6.3 Laboratory Instruments

All laboratories conducting analyses of samples collected under the contract are required to have a preventative maintenance program covering testing, inspection, and maintenance procedures and schedule for each measurement system and required support activity. This program is usually documented in the form of SOPs for each analytical instrument to be used. The basic requirements and components of such a program include the following:

- Each laboratory will have, as a part of its QA/QC program, a routine preventive maintenance program conducted to minimize the occurrence of instrument failure and other system malfunction.
- Service and repair of instruments, equipment, tools, gauges, and so forth will be performed by an internal group of qualified personnel. Alternatively, scheduled instrument maintenance

and emergency repair may be provided by manufacturers' representatives under a repair and maintenance contract.

- Instrument maintenance will be carried out by the laboratory on a regularly scheduled basis. The servicing of critical items should be scheduled to minimize the downtime of the measurement system. A list of critical spare parts for each instrument will be identified by the laboratory and requested from the manufacturer. These spare parts will be stored at the laboratory for availability and use to reduce downtime. The availability of spare parts will be monitored periodically.
- Testing, inspection, and maintenance procedures described in laboratory SOPs will be in accordance with manufacturer's specifications and with the requirements of the specific analytical methods employed.
- All maintenance and service must be documented in service logbooks to provide a history of maintenance records. A separate service logbook should be kept for each instrument. All maintenance records will be traceable to the specific instrument, equipment, tool, or gauge.
- Records produced as a result of testing, inspection, or maintenance of laboratory instruments will be maintained and filed at the laboratory. These records will be available for review by internal and external laboratory system audits under the contract.

3.7 Instrument and Equipment Calibration and Frequency

Instruments will be calibrated according to manufactures specifications. Field instruments will be calibrated prior to each sampling event or as instructed by the manufacturer. Field instruments include but not limited to temperature, pH and conductivity meter, and photo ionization detector.

This section describes the procedures for maintaining the accuracy of field equipment and laboratory instruments used for field tests and laboratory analyses. The equipment and instruments should be calibrated before each use or on a scheduled, periodic basis when not in use.

3.7.1 Field Equipment

Equipment used to collect field samples or take field measurements under the contract will be maintained and calibrated with sufficient frequency and in such a manner that the accuracy and reproducibility of results are consistent with the manufacturer's specifications and with project-specific DQOs.

The contractor field team leader is to verify that field sampling and measurement equipment is in good working condition. The manufacturer's operating manual and instructions that accompany the equipment will be consulted to ensure that all calibration procedures are followed.

Field measurements will vary according to project requirements. Project work plans and/or QA/QC documents will identify the types of field equipment to be used, identify the equipment requiring calibration, and include SOPs covering equipment calibration procedures, requirements for calibration standards and apparatus, calibration frequencies, and requirements for maintaining calibration records and traceability. The project work plan and/or QA/QC documents will also discuss any unique, project-specific calibration requirements.

3.7.2 Laboratory Instruments

All laboratory equipment used to analyze samples collected under the contract will be calibrated based upon written SOPs maintained by the laboratory. Calibration records (including the dates and times calibration and the names of the personnel performing the calibration) will be filed at the location where the analytical work is performed and maintained by the laboratory personnel performing QC activities. Calibration records will be subject to QA audits. Most laboratory work under the contract will be

conducted by subcontractor laboratories. In all cases, the laboratory subcontractor QA manager is responsible for ensuring that all laboratory instruments are calibrated in accordance with the requirements in this Generic QAPP and in any site-specific QAPP Addendum or project-specific QAPP.

Because laboratory analytical methods will vary with each project, specific calibration procedures cannot be addressed in this Generic QAPP. However, the project work plan with site-specific R7 QAPP Addendum Form will reference the method's calibration procedures and requirements for all laboratory measurements. Calibration procedures and requirements will also be provided as appropriate for laboratory support equipment such as balances, mercury thermometers, pH meters, and other equipment used to make chemical and physical measurements.

When analyses are conducted in accordance with SW-846 methods, calibration procedures and frequencies specified in the relevant method should be followed as closely as possible. The site-/project-specific QA/QC documents should provide any additional calibration requirements (such as equipment requiring calibration, calibration procedures, requirements for calibration standards and apparatus, requirements for maintaining calibration records and traceability, calibration frequency, acceptance criteria, number of calibration points, and internal or external standards) that deviate from or are not specified in the published EPA-approved method. Such deviations will be outlined in the site-/project-specific QA/QC documents or in an appendix as part of a laboratory SOP.

For analytical methods that are not EPA-approved, a complete SOP including the calibration procedures for the method will be included as an appendix to the appropriate project QA/QC document. Laboratory SOPs describing calibration procedures for such non-standard methods should include the following information:

- Detailed calibration procedure for each instrument used.
- Internal standard or external standard calibration requirements and procedures.
- Calibration requirements for confirmatory results (second column, second detector, mass spectral confirmation, and so forth).
- Frequency of calibration and continuing calibration checks.
- Number of calibration standards used, concentrations, and preparation methods.
- Traceability of calibration standards and continuing calibration check standards.
- Numerical acceptance criteria for initial calibration and continuing calibration checks.
- Corrective action procedures for situations where calibration procedures are not performed properly, or calibration acceptance criteria are not met.
- Instructions for recording calibration information and results, including what information is to be recorded and where it is recorded and stored.

3.8 Inspection and Acceptance Requirements for Supplies and Consumables

Contractor's Project Managers have primary responsibility for identifying the types and quantities of supplies and consumables needed for environmental data collection projects conducted under the contract. Contractor's Project Managers are also responsible for determining acceptance criteria for these items. The contractor's Project Manager will ensure that any required certification is in place and document this in the field notebook and in the report prepared for EPA.

Supplies and consumables can be received either at a contractor office or at a site. When supplies are received at a contractor office, the contractor project manager or contractor field team leader will sort the supplies according to vendor, check packing slips against purchase orders, and inspect the condition of all supplies before the supplies are accepted for use on a project. If the supplies do not meet the acceptance

criteria, deficiencies will be noted on the packing slip and purchase order. In addition, a form will be completed describing the problem and circumstances in full, and noting the purchase order number for the item. The item will then be returned to the vendor for replacement or repair.

Procedures for receiving supplies and consumables in the field are similar to those described above. Upon receipt, items will be inspected by the contractor's Project Manager or field team leader against the acceptance criteria. Any deficiencies or problems will be noted in the field logbook, and deficient items will be returned for immediate replacement.

3.9 Non-Direct Measurements (Data Acquisition) Requirements

Previous investigations and sampling data acquired by EPA, State Environmental Agencies, other Federal Agencies or its contractor were all subject to Quality Assurance Project Plans and other quality controls. This information was used to select the sites for the site assessment activities for which this Generic QAPP was prepared. Previously acquired sampling data will be included in the site assessment reports, as well as the source of this data.

Some work conducted under the contract may not involve direct measurement. This includes activities that use data drawn from other sources such as databases, spreadsheets, and literature files. When such data is of critical importance in supporting sampling and analytical measurements, QA requirements for the non-direct measurement will be outlined in the project work plan with SAP and site-specific QAPP Addendum or project-specific QAPP. The source and quality of the data, along with potential problems affecting its applicability or limitations, will be documented. Such data will be reviewed for quality and supporting documentation. If supporting documentation does not accompany the data, a records or file search will be conducted to obtain the supporting documentation. Supporting documentation will be used in part to evaluate the quality and usefulness of the data. For example, if historical sampling data are to be used for an activity, the data should be reviewed to determine the QA procedures that were implemented. If such information is not available, the use of the data will be limited. Generally, data that are not supported by documentation of acceptable procedures cannot be used for enforcement purposes but may be useful for preliminary analysis and assessment. In all cases, evaluation and verification procedures for non-direct measurement data should be approved by the contractor's Quality Assurance Manager or his designee.

When a large external data set is used, computer-assisted data screening will be applied to determine the internal consistency of the data set. The goal of such screening is to identify outliers from the overall data set. When data accuracy is primarily an issue of transcription accuracy, such as keying large data sets into a computer file, proof readers will perform checks independent of the computer-assisted data screening.

3.10 Data Management Requirements

The following paragraphs provide general discussion and requirements for managing data under the contract for EPA. Further detail and requirements will be provided as necessary in the site-/project-specific QA/QC documents, including requirements for data recording, validation, transformation, transmittal, reduction, analysis, tracking, storage, and retrieval. The site-/project-specific QA/QC documents will also provide, as necessary, checklists and standard forms for detecting and correcting errors and preventing the loss of data during data reduction, data reporting, data encoding, and data entry.

Data for the contract will be obtained from a combination of sources, including field measurements and analyses, and subcontractor laboratories. The process of data gathering is a coordination effort and will be conducted by project staff in conjunction with all potential data producers. The data itself will be obtained from the analytical service provider, when appropriate, in the form of an electronic data deliverable in addition to the required hard copy analytical data package. The standard data management software of all analytical data to be submitted electronically by the contractor is SCRIBE. A hardcopy of

the data will also be required as part of the site assessment report. The EPA Project Manager will review the data to ensure accuracy prior to placing into the facility file.

Data tracking is imperative to ensure timely, cost-effective, and high-quality results. Data tracking begins with sample chain-of-custody. When the analytical services provider receives the samples into custody, the provider will send a sample acknowledgment to the contractor. The sample acknowledgment will confirm the sample receipt, condition, and the required analyses.

Unless otherwise directed by EPA, the contractor will validate all data generated under the contract as described in Section 5.2 of this Generic QAPP. As a part of the data validation process, the electronic data deliverables will be reviewed against the hard copy deliverables to ensure accurate transfer of data. In addition, the hard copy will be evaluated for errors in calculation of results. As a result of the data validation, qualifiers will be placed on the data to indicate the data usability. These qualifiers will be placed into the electronic data file. Upon approval of the data set with the appropriate data qualifiers, the electronic data will be released to the project leader for reporting. A complete discussion of data validation procedures is contained in Sections 5.1 and 5.2 of this Generic QAPP.
Following data validation and release of data, the contractor project managers will use data to prepare project reports. As a part of the final report quality control review procedures, the data will be further checked by technical reviewers and a Quality Control Coordinator (QCC) to verify its accuracy in the report.

In addition to the final report, all analytical data in the form obtained from the analytical services provider will be archived with the final project file in a secure location. The secure location will house all final project files until they are transferred to EPA.

4.0 ASSESSMENT AND OVERSIGHT

This section of the Generic QAPP includes the two QAPP elements required by EPA QA/R-5 (EPA, 2002a) to assess and evaluate the management of environmental data collection operations. These QAPP elements provide procedures for conducting appropriate audits and reports and implementing corrective actions as necessary to ensure that the quality of data generated by implementation of this Generic QAPP is adequate. The two QAPP elements related to assessment and oversight are:

- Assessment and response actions (Section 4.1).
- Reports to management (Section 4.2).

4.1 Assessment and Response Actions

The EPA Regional Quality Assurance Manager and Project Managers will evaluate the process and quality of performance on a site-by-site basis. All measured parameters will be observed to ensure that the data meets the QA/QC requirements and other site-specific requirements identified in the TO or PR and/or project work plan documents. Every attempt will be made to subcontract analytical work to a National Environmental Laboratory Accreditation Program (NELAP) certified laboratory. If a non-NELAP certified commercial laboratory is used, assessment and response of the analytical phases will be in accordance with that laboratory's internal QA procedures. When a non-NELAP laboratory is used, deviations to EPA SOP 2440.05C will be documented in the site-specific QAPP Addendum or project-specific QAPP. The contractors/subcontractors will provide copies of the SOPs for the mobile and fixed laboratory, as part of the contractual agreements and conditions of this Generic QAPP, before providing any actual site assessment sampling services.

The EPA Project Manager may periodically visit the site to observe the field activities and determine whether field personnel are following the project work plan with SAP, the site-specific QAPP Addendum or project-specific QAPP, and SOPs, and to take corrective action if necessary. This should be documented in the field report as well as the site assessment reports. The program Generic QAPP will be revised if necessary, to ensure that program and appropriate QA/QC objectives and requirements are being achieved.

Under the contract, performance and system audits of both field and laboratory activities may be conducted to verify that sampling and analysis are performed in accordance with the procedures and requirements established in this Generic QAPP, project work plans, site-specific QAPP Addendum or project-specific QAPP. Non-conforming items identified during an audit will be addressed by corrective action. This section addresses basic audit and corrective action requirements that apply to all work conducted by the contractor under the contract. If additional project-specific audits are required by a TO or PR, they will be identified in the site-specific QAPP Addendum or project-specific QAPP.

4.1.1 Performance and System Audits

Both internal performance and system audits may be conducted on the contractor's field operations and subcontractor laboratories under the contract. Performance audits include verification that field sampling activities and measurements and laboratory analyses of performance evaluation samples are being conducted in accordance with the requirements of this Generic QAPP and any site-specific QAPP Addendum or project-specific QAPP. System audits involve a qualitative examination of all components of an environmental data collection system, including records, personnel, and QA management activities.

This section describes the selection of audit personnel, the scope of field and laboratory audits, audit frequencies, and typical audit reports for internal audits initiated by contractor's Quality Assurance Manager. External performance and system audits initiated by EPA may also be conducted under the contract and would involve similar activities.

4.1.1.1 Audit Personnel

All auditors must be independent of the activities being audited. The contractor's Quality Assurance Manager has the lead role in directing all internal audit activities during an investigation. The contractor's Quality Assurance Manager will select the appropriate personnel to conduct each internal audit and will assign them responsibilities and deadlines for completing their audits. These personnel may include the contractor's Quality Assurance Manager, or other independent auditors. When an audit team is required, the contractor's Quality Assurance Manager selects a lead auditor based on relevant technical expertise and audit experience. The lead auditor is responsible for selecting and preparing the audit team; preparing an audit plan; coordinating and scheduling the audit with the project team, subcontractor, or other organization being audited; participating in the audit; coordinating the preparation and issuance of audit reports and corrective action request forms; and evaluating audit responses and resulting corrective actions.

4.1.1.2 Audit Scope of Work

Performance audits of field activities will be conducted to evaluate compliance with the requirements of this Generic QAPP, site-specific QAPP Addendum or project-specific QAPP, and any applicable work plan/SAP with R7 QAPP Addendum Form, and SOP documents. Field systems audits may include an examination of the following items:

- Sample collection records.
- Sample collection, handling, preservation, packaging, shipping, and custody records.
- Equipment operation, maintenance, and calibration records.

Laboratory performance audits include analysis of blind performance evaluation samples to assess a laboratory's ability to comply with QC control limits. Laboratory systems audits may include evaluation of the following:

- Sample log-in, identification, storage, tracking, and custody procedures.
- Sample and standards preparation procedures.
- Availability of analytical instruments.
- Analytical instrument operation, maintenance, and calibration records.
- Laboratory security procedures.
- Qualifications of analysts.
- Case file organization and data handling procedures.

4.1.1.3 Audit Frequencies

As necessary, the site-/project-specific QA/QC documents will provide a schedule of all planned audits that will be conducted during the investigation. These audits may be required by EPA or planned by the contractor's Quality Assurance Manager. Audit frequency will depend on several factors. In selecting projects for auditing, the contractor's Quality Assurance Manager will consider projects with a large volume of work or those on which EPA has placed a high level of importance. The contractor's Quality Assurance Manager may also randomly select projects for auditing. For laboratory audits, the contractor's Quality Assurance Manager will focus on laboratories performing critical measurements (as determined by DQOs) and on subcontractor laboratories performing work for the first time.

Unscheduled follow-up audits may occur if any deficiencies are discovered during an audit or review. Follow-up audits serve to ensure that all necessary corrective actions have been properly implemented to address deficiencies.

4.1.1.4 Audit Reports

Audit reports will be prepared for performance and system audits of field and laboratory activities and all laboratory performance evaluation studies that are conducted under the contract. Reports will be prepared by the lead auditor responsible for coordinating the audit. Audit reports will identify audit participants, describe the activity audited, summarize audit findings, and detail any deficiencies or deviations from protocol that were discovered during the audits, as well as any corrective actions that are proposed. Any field or laboratory analytical data that is generated during the analysis of blind performance evaluation samples must be validated. The validated data will be included with the audit report. Data validation procedures are discussed in Section 5.2.

Audit reports are distributed to the contractor's Quality Assurance Manager, Contractor Administrator, contractor's Project Manager, and the Field Team Leader or the laboratory subcontractor's Quality Assurance Manager, as appropriate. The lead auditor has primary responsibility for ensuring that audits are conducted thoroughly and properly. Contractor's Project Managers and team field or laboratory subcontractor's Quality Assurance Manager are responsible for implementing corrective actions that result from an audit. The contractor's Quality Assurance Manager is responsible for verifying that recommended corrective actions have been implemented.

4.2.1 Corrective Action

Rapid and thorough correction of QA problems, through an effective corrective action program, minimizes the possibility of questionable data or documentation. The two types of corrective action are immediate and long-term. Immediate corrective actions include correcting procedures, repairing instruments that are working improperly, and correcting errors or deficiencies in documentation. Long-term corrective actions eliminate the sources of problems by correcting systematic errors in sampling and analytical procedures, replacing procedures that produce questionable results, and manipulating similar cause-and-effect relationships.

All QA problems and corrective actions applied are documented to provide a complete record of QA activities. These records assist the contract administrator management team in identifying long-term QA problems and enable application of long-term corrective actions such as personnel training, replacement of instruments, and improvement of sampling and analytical procedures.

The contractor's Quality Assurance Manager has the authority to discontinue or limit environmental data measurements that are compromised until corrective action is complete and data quality is no longer questionable. The contractor's Quality Assurance Manager may also order the recollection or reanalysis of samples or remeasurement of field parameters since the last documented evidence that the measurement system was in control.
Specific corrective action procedures for sample collection and field measurements and laboratory analyses are discussed below.

4.1.2.1 Sample Collection and Field Measurements

Technical staff and project personnel involved in sample collection or field measurement activities are responsible for initiating routine corrective actions by reporting all suspected technical or QA nonconformance's and deficiencies to the contractor project manager or his/her designee. Corrective actions for sample collection and field measurements may include, but are not limited to, the following:

- Repeating measurements to check for error.

- Checking that instruments are properly adjusted for ambient conditions such as temperature.
- Checking batteries.
- Checking calibration and recalibrating equipment if necessary.
- Replacing the instrument or measurement devices.
- Collecting additional samples.
- Stopping work (if necessary).

4.1.2.2 Laboratory Analyses

Each laboratory that participates as a subcontractor is required to write a SOP summarizing procedures for initiating, developing, approving, implementing, and documenting corrective action. The existence of such a program does not exempt the laboratory from following the corrective action requirements outlined in this Generic QAPP or in any site-specific QAPP Addendum or project-specific QAPP. When errors, deficiencies, or out-of-control situations arise, systematic corrective actions must be taken to resolve problems and restore proper functioning analytical systems. Laboratory personnel and Quality Assurance Managers are alerted that corrective actions may be necessary if any of the following situations arise:

- Sample volumes are not sufficient to perform required analyses.
- QC data are outside the acceptable limits for precision and accuracy.
- Blanks contain contaminants above acceptable levels.
- Undesirable trends are detected in spike recoveries or in the RPD between duplicates.
- Unusual changes in detection limits arise.
- Deficiencies are detected during internal or external audits or from the results of performance evaluation samples.
- Inquiries concerning data quality are received from clients.

If sample volumes are insufficient to complete the required analyses, the laboratory will notify the contractor project manager. The contractor's Project Manager, contractor's Quality Assurance Manager, and laboratory subcontractor's Quality Assurance Manager will contact the EPA Project Manager to determine if additional samples need to be collected.

Laboratory corrective action procedures are often initiated at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors; checks the instrument calibration; checks the spiking levels, calibration solutions, and standards; and checks instrument sensitivity. If the problem persists or cannot be identified, the matter may be referred to the laboratory supervisor, Project Manager, or Quality Assurance Manager for further investigation. Every effort must be made to determine the cause of the problem so that a permanent solution can be developed and implemented. Once a problem is resolved, full documentation of the corrective action procedure is filed with the project records.

Investigations initiated by laboratory technical or Quality Assurance personnel that result in corrective actions must be documented and reported to the contractor's Quality Assurance Manager. Documentation of investigations of negative performance on performance evaluation samples and corrective actions taken will be forwarded to the appropriate certifying agencies when required.

4.2 Reports to Management

A draft report for Superfund's SA, ISA, RSE or RI projects will be prepared by the EPA contractor at the completion of the field sampling effort and upon receipt of validated laboratory data. The report will inform the EPA Project Manager of the status of the project; results of performance evaluations and

system audits; results of periodic data quality assessments; and significant quality assurance problems and recommended solutions.

Following review by the EPA Project Manager, the EPA contractor will prepare a final report to be incorporated into the SA, ISA, RSE or RI reports for submission to EPA Region 7, as appropriate.

Effective management of environmental data collection operations requires timely assessment and review of measurement activities. Open communication, interaction, and feedback must also occur among all project participants, including contractor's corporate Quality Assurance Manager, the EPA Quality Assurance Manager or a designated representative, contractor's Contract Administrator, contractor's Project Manager, contractor's Quality Assurance Manager, technical staff, and team subcontractors.

5.0 DATA VALIDATION AND USABILITY

This section of the Generic QAPP includes the three QAPP elements required by EPA QA/R-5 (2001a) to ensure that data is valid and usable for its intended purpose. The three QAPP elements related to data validation and usability are:

- Data review, verification, and validation requirements (Section 5.1).
- Validation and verification methods (Section 5.2).
- Reconciliation with data user requirements (data quality objectives) (Section 5.3).

5.1 Data Review, Verification, and Validation Requirements

Data review and verification will be performed by a qualified laboratory analyst as described in the mobile and fixed laboratory SOPs (as described in Sections 3.0 and 4.0 above). The SOPs from the mobile and fixed laboratories will be added as an addendum to the QAPP. The EPA Project Manager will be responsible for the validation and final approval of the data (including field notes) in accordance with the stated project purpose and use of the data. Any anomalies will be documented with corrective actions described and included in the site assessment report.

This section focuses on data review and reduction requirements for work conducted under the contract. Data validation and verification requirements are covered in Section 5.2.

Data reduction and review are essential functions for preparing data that can be effectively used to support project decisions and DQOs. These functions must be performed accurately and according to EPA-approved procedures and techniques and region-specific guidelines (ESDOQAM). Data reduction includes all computations and data manipulations that produce the final results used during the investigation. Data review includes all procedures conducted by field or laboratory personnel to ensure that measurement results are correct and acceptable relative to QA objectives in this Generic QAPP and in any site-specific QAPP Addendum or project-specific QAPP.

Because the types of field measurements and laboratory measurements used will vary with each site investigation, most data reduction and review procedures and requirements cannot be addressed directly in this Generic QAPP. However, many field and laboratory measurement data reduction and review procedures and requirements are specified in field and laboratory methods, SOPs, and guidance documents. In most cases, data review and reduction procedures can be identified in the site-/project-specific QA/QC documents or in a site-specific QAPP Addendum or project-specific QAPP by referencing these sources. However, if data review and reduction are not adequately described in these sources, the site-/project-specific QA/QC documents or the site-specific QAPP Addendum or project-specific QAPP should include the following information:

- Outlined data review and reduction procedures for all phases of sample preparation and analysis (including procedures for data that are reduced and stored on computer).
- Field personnel and laboratory personnel responsible for conducting each phase of data review and reduction.
- All formulas and equations used during data reduction, including all equations used to produce final results.
- The definitions of all terms and parameters.
- The units for all parameters and results.
- Instructions on how the results from QC samples (such as blanks) will be treated and used in calculating the final results.

- Procedures for flagging, qualifying, or marking the data with labels.
- Corrective action procedures for instances when data reduction procedures are not followed correctly or when errors are found during data review.

Field personnel will record all raw data from chemical and physical field measurements in a field logbook. Contractor's Project Managers have primary responsibility for (1) verifying that field measurements were made correctly, (2) confirming that sample collection and handling procedures specified in the site-/project-specific QA/QC documents were followed, and (3) ensuring that all field data reduction and review procedures and requirements are followed. They are also responsible for assessing preliminary data quality and for advising the data user of any potential QA/QC problems with field data. When field data are used in a project report, data reduction methods will be fully documented in the report.

Each laboratory subcontractor will complete data reduction for chemical and physical laboratory measurements and will complete an in-house review of all laboratory analytical results. The laboratory subcontractor's Quality Assurance Manager is responsible for ensuring that all laboratory data reduction and review procedures and requirements in this Generic QAPP and/or in the site-specific QAPP Addendum or project-specific QAPP are followed. The laboratory subcontractor's Quality Assurance Manager is also responsible for assessing data quality and for advising the contractor's Quality Assurance Manager of possible QA/QC problems with laboratory data.

5.2 Verification and Validation Methods

The data will be validated in accordance with the mobile and fixed laboratory SOPs (see above). Field notes will be compared for consistency and the EPA Project Manager will document any anomalies. The EPA Project Manager will inspect the data to provide final review and approval to ensure that the data meets the sampling requirements.

All data that are used to support activities under the contract must be valid for their intended purposes. This section outlines the basic data validation procedures that will be followed for all field and laboratory measurements. The following subsections identify personnel responsible for data validation and the general data validation process and EPA data validation guidance that will be followed.

5.2.1 Data Validation Responsibilities

The contractor's Quality Assurance Manager, or his/her designee, is responsible for validating all field and laboratory data collected under the contract. The laboratory subcontractor will also validate all laboratory data according to their own specific procedures before submitting the data to the contractor. As requested the contractor will validate all laboratory subcontractor data, unless specified otherwise in the work plan or approved by the EPA Project Manager. Data validation will be completed by one or more experienced data reviewers. When applicable, site-specific QAPPs will include the names and qualifications of data reviewers assigned to the project.

5.2.2 Data Validation Procedures

The validity of a set of data is determined by comparing the data with a predetermined set of QC limits. For investigations conducted under the contract, these QC limits will be provided or referenced in each project-specific study. Contractor data reviewers will conduct a systematic review of the data for compliance with established QC limits (for example, sensitivity, precision, and accuracy) based on spike, duplicate, and blank sample results provided by the laboratory. The data review will identify any out-of-control data points or omissions. Contractor data reviewers will evaluate laboratory data for compliance with the following:

- Method and project-specific analytical service requests.
- Holding times.

- Initial and continuing calibration acceptance criteria.
- Field, trip, and method blank acceptance criteria.
- Surrogate recovery.
- Field duplicates, MS/MSD and matrix duplicate acceptance criteria.
- Other laboratory QC criteria specified by the method and the project-specific analytical service request.
- Compound identification and quantitation.
- Overall assessment of data in accordance with project-specific objectives.

The contractor will follow the most current EPA guidelines for completing data validation:

- "Data Validation Standard Operating Procedures for Contract Laboratory Program Routine Analytical Services. Revision 2.1." U.S. EPA Region 7. Science and Ecosystem Support Division. Office of Quality Assurance. (EPA, 1999a).
- "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review." Publication 9240.1-05-01. (EPA, 1994a).

General procedures in the EPA guidelines will be modified as necessary to fit the specific analytical method used to produce the data.

In all cases, data validation requirements will depend on DQO levels, region-specific guidelines, reporting requirements and data deliverables requested from the laboratory. Data validation requirements presented in these sections may be referenced in the work plan/SAP, or site-specific QAPP Addendum or project-specific QAPP to ensure consistency.

5.3 Reconciliation with User Requirements (Data Quality Objectives)

The EPA Project Manager, for completeness needed to achieve the project's goal, will evaluate data. If the data quality indicators do not meet the project requirements outlined in the site-specific QAPP Addendum or project-specific QAPP, the data may be discarded and re-sampling may occur. In case of a failure, the project team will evaluate the cause. If the failure is due to laboratory procedures or equipment, necessary corrective measures will be taken by the EPA Quality Assurance Manager and EPA Project Manager. If failure is associated with sampling, field procedures will be re-evaluated with any changes documented by the EPA Project Manager and included in the site assessment report.

The primary purpose of a QA system is to define a process for collecting data that is of known quality, is scientifically valid, is legally defensible, and fully supports any decisions that will be based on the data. To achieve this purpose, this Generic QAPP requires that DQOs be fully defined in Section 2.5. All other parts of the QA system must then be planned and implemented in a manner consistent with the DQOs. The QA system components that follow directly from the DQOs include documentation and reporting requirements (Section 2.8); sample network design and sampling methods (Sections 3.1 and 3.2); analytical methods requirements (Section 3.4); QC requirements (Section 3.5); and data reduction, validation, and reporting methods (Sections 5.1 and 5.2).

Once environmental data have been collected, reviewed, and validated, the data must be further evaluated to determine whether the DQOs identified in the project work plan/SAP, the site-specific QAPP Addendum, or the project-specific QAPP have been met. Contractor will follow EPA's data quality assessment (DQA) process to verify that the type, quality, and quantity of data collected are appropriate for their intended use. The DQA process involves first verifying that the assumptions under which the data collection design and DQOs were developed have been met, or taking appropriate corrective action if the assumptions have not been met. The DQA process then evaluates how well the data collected support

the decision that must be made so that scientifically valid and meaningful conclusions can be drawn from the data. To the extent possible, Contractor will follow DQA methods and procedures outlined in EPA documents Data Quality Assessment: A Reviewer's Guide QA/G-9R (EPA, 2006c) and Data Quality Assessment: Statistical Tools for Practitioners QA/G-9S (EPA, 2006d).

If data quality indicators do not meet the project's requirements as outlined in the QAPP, the data may be discarded, and re-sampling and/or re-analysis may be required.

REFERENCES

American Society for Testing and Materials (ASTM). 1998. *Standard Test Method for Particle-Size Analysis of Soils*. D 422-63. 1998.

American National Standards Institute/American Society for Quality Control (ANSI/ASQC). *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (E4-119).* 1995. American National Standard.

American Society for Testing and Materials (ASTM). *Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives*. D5792-95. January 1996.

American Society for Testing and Materials (ASTM). 2005. *Standard Practice for Collection of Floor Dust for Chemical Analysis.* Annual Book of ASTM Standards. ASTM D 5438-05. 2005.

CS_3, Inc. 1998. *High Volume Small Surface Sampler, HVS3, Operation Manual*. 1998.

National Institute for Occupational Safety and Health (NIOSH). *NIOSH Manual of Analytical Methods, Fourth Edition.* 1994. Publication No. 94-113.

1991 U.S. EPA

U.S. Environmental Protection Agency (EPA). *Management of Investigation-Derived Wastes During Site Inspections*. May. OERR Directive 9345.3-02. EPA Publication EPA/540/G-91/009. 1991a.

U.S. Environmental Protection Agency (EPA). *Compendium of ERT Soil Sampling and Surface Geophysics Procedures.* January. OSWER Publication 9360.4-02. EPA Publication EPA/540/P-91/006. 1991b.

U.S. Environmental Protection Agency (EPA). *Compendium of ERT Surface Water and Sediment Sampling Procedures.* January. OSWER Publication 9360.4-03. EPA/540/P-91/005. 1991c.

U.S. Environmental Protection Agency (EPA). *Compendium of ERT Ground Water Sampling Procedures.* January. OSWER Publication 9360.4-06. EPA Publication EPA/540/P-91/007. 1991d.

U.S. Environmental Protection Agency (EPA). *Compendium of ERT Waste Sampling Procedures.* January. OSWER Publication 9360.4-07. EPA Publication EPA/540/P-91/008. 1991e.

1992 U.S. EPA

U.S. Environmental Protection Agency (EPA). *Compendium of ERT Air Sampling Procedures.* May. OSWER Publication 9360.4-05. 1992a.

U.S. Environmental Protection Agency (EPA). *Compendium of ERT Field Analytical Procedures*. May. OSWER Directive 9360.4-04. 1992b.

1994 U.S. EPA

U.S. Environmental Protection Agency (EPA). *U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review.* February. U.S. Environmental Protection Agency. Office of Emergency and Remedial Response. Washington, DC. EPA Publication 9240.1-05-01. 1994a.

U.S. Environmental Protection Agency (EPA). *Guidance Manual for the IEUBK Model for Lead in Children*. February. OSWER 9285.7-15-1. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. Washington, DC. EPA Publication 93-963510. 1994b.

1995 U.S. EPA

U.S. Environmental Protection Agency (EPA). *Superfund Program Representative Sampling Guidance Volume 1: Soil*. December. OSWER Directive 9360.4-10. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. EPA Publication EPA/540/R-95/141. 1995a.

U.S. Environmental Protection Agency (EPA). *Residential Sampling for Lead: Protocols for Dust and Soil Sampling*. March. U.S. Environmental Protection Agency. Pollution Prevention and Toxics (7404). EPA Publication 747-R-95-001. 1995b.

1996 U.S. EPA

U.S. Environmental Protection Agency (EPA). *Test Methods for Evaluating Solid Waste, Volumes IA through IC, SW-846, Third Edition.* December. Update III. Office of Solid Waste and Emergency Response. Washington, DC.

1999 U.S. EPA

U.S. Environmental Protection Agency (EPA). *Data Validation Standard Operating Procedures for Contract Laboratory Program Routine Analytical Services.* July. Revision 2.1. U.S. EPA Region 7. Science and Ecosystem Support Division. Office of Quality Assurance. 1999a.

U.S. Environmental Protection Agency (EPA). *U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review.* October. Office of Emergency and Remedial Response. Washington, DC. EPA Publication 9240.1-05A-P. 1999b.

2000 U.S. EPA

U.S. Environmental Protection Agency (EPA). *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (ESDOQAM).* January. Region 7 Environmental Services Division. 2000a.

U.S. Environmental Protection Agency (EPA). *Guidance for the Data Quality Objectives Process for Hazardous Waste Site Investigations.* EPA QA/G-4HW. January. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. 2000b.

U.S. Environmental Protection Agency (EPA). *Short Sheet: TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites*. April. OSWER Publication 9285.7-38. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. EPA Publication EPA/540-F-00-010. 2000c.

2001 U.S. EPA

U.S. Environmental Protection Agency (EPA). *EPA Requirements for Quality Assurance Project Plans*. EPA QA/R-5. March. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/B-01/003. 2001a

U.S. Environmental Protection Agency (EPA). *Using the Triad Approach to Improve the Cost-Effectiveness of Hazardous Waste Site Cleanup*. OSWER Publication 542-R-01-016.

U.S. Environmental Protection Agency (EPA). *EPA Requirements for Quality Management Plans*. March EPA QA/R-2. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/B-01/002.

U.S. Environmental Protection Agency (EPA). *Guidance for Preparing Standard Operating Procedures (SOPs)*. EPA QA/G-6. March. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/B-01/004.

U.S. Environmental Protection Agency (EPA). *Data Quality Objectives Decision Error Feasibility Trials Software (DEFT)-User's Guide*. EPA QA/G-4D. September. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/B-01/007.

2002 U.S. EPA

U.S. Environmental Protection Agency (EPA). *Guidance for Quality Assurance Project Plans.* EPA QA/G-5. December. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/R-02/009. 2002a.

U.S. Environmental Protection Agency (EPA). *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan*. EPA QA/G-5S. December. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/R-02/005. 2002b.

U.S. Environmental Protection Agency (EPA). *User's Guide for the Integrated Exposure Uptake Biokinetic Model for Lead in Children (IEUBK) Windows® Version-32 Bit Version*. OSWER 9285.7-42. May. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. EPA Publication 540-K-01-005. 2002c.

U.S. Environmental Protection Agency (EPA). *Guidance on Environmental Data Verification and Data Validation*. EPA QA/G-8. November. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/R-02/004.

U.S. Environmental Protection Agency (EPA). *Guidance for Geospatial Data Quality Assurance Project Plans*. EPA QA/G-5G. December. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/R-02/009.

2003 U.S. EPA

U.S. Environmental Protection Agency (EPA). *Superfund Lead-Contaminated Residential Sites Handbook*. OSWER 9285.7-50. August. U.S. Environmental Protection Agency. Office of Emergency and Remedial Response. 2003.

2004 U.S. EPA

U.S. Environmental Protection Agency (EPA). Superfund Analytical Services/Contract Laboratory Program Website. Online address: http://www.epa.gov/superfund/programs/clp/index.htm.

2006 U.S. EPA

U.S. Environmental Protection Agency (EPA). *Guidance on Systematic Planning Using the Data Quality Objectives Process*. EPA QA/G-4. February. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/B-06/001. 2006a.

U.S. Environmental Protection Agency (EPA). *Systematic Planning: A Case Study for Hazardous Waste Site Investigation*. EPA QA/CS-1. February. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. 2006b.

U.S. Environmental Protection Agency (EPA). *Data Quality Assessment: A Reviewer's Guide*. EPA QA/G-9R. February. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/B-06/002. 2006c.

U.S. Environmental Protection Agency (EPA). *Data Quality Assessment: Statistical Tools for Practitioners*. February. U.S. Environmental Protection Agency. Office of Environmental Information. Washington, DC. EPA Publication EPA/240/B-06/003. 2006d.

U.S. Environmental Protection Agency (EPA). Region 7. *Quality Management Plan for Region 7*. August.

APPENDICES

APPENDIX A

REGION 7
QUALITY ASSURANCE PROJECT PLAN FORM
(R7 QAPP FORM)

Region 7 Superfund Program
Quality Assurance Project Plan Form

Project Information:

Site Name: Cherokee County Superfund Site OU 8 (TO73)			**City:** Cherokee County (County-Wide)	**State:** Kansas
EPA Project Manager: Elizabeth Hagenmaier			**HGL Project Manager:** Andrea Fletcher	
Approved By:			**Prepared For:** EPA Region 7 Superfund Division	
Title:	HGL Project Manager	**Date:**		
Approved By:				
Title:	HGL Program Manager	**Date:**	**Prepared By:** Andrea Fletcher	
Approved By:				
Title:	HGL QA Manager	**Date:**	**Date:** March 31, 2017	
Approved By:				
Title:	EPA Project Manager	**Date:**	**AES Contractor:** HydroGeoLogic, Inc. (HGL)	
Approved By:			**AES Contract Number:** EP-S7-05-05	
Title:	EPA Superfund QA Coordinator	**Date:**		

1.0 Project Management:

1.1 Distribution List

EPA--Region 7: Elizabeth Hagenmaier, EPA Project Manager
Diane Harris, Superfund QA Coordinator

AES Project Managers: Andrea Fletcher

1.2 Project/Task Organization

Elizabeth Hagenmaier, the EPA TOPO, will serve as the EPA Project Manager for activities described in this QAPP.
Andrea Fletcher from HGL will serve as the Task Order Manager (TOM), the HGL Project Manager.
Personnel for soil sampling.

1.3 Problem Definition/Background:

Description: This site-specific Quality Assurance Project Plan form is prepared as an addendum to the **Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites (June 2007)** and contains site-specific data quality objectives (Table 2) for the sampling activities described herein.

☐ Problem Definition/Background Description attached.

☒ Description in referenced report: Final RI Report Cherokee County Operable Unit 8 Railroads Site (**Title**) March 2016 (**Date**)

1.4 Project/Task Description:

☐ CERCLA PA ☐ CERCLA SI ☐ Brownfields Assessment
☒ **Other (description included)** ☐ Pre-CERCLIS Site Screening ☐ Removal Assessment

Schedule: Field work is tentatively scheduled for May - June 2017.

☐ Description in referenced report: _____ (**Title**) _____ (**Date**)

1.5 Quality Objectives and Criteria for Measurement Data:

a. Accuracy: ☒ Identified in attached Table 2.
b. Precision: ☒ Identified in attached Table 2.
c. Representativeness: ☒ Identified in attached Table 2.
d. Completeness: ☒ Identified in attached Table 2.
e. Comparability: ☒ Identified in attached Table 2.

Other Description: A completeness goal of 100 percent for the confirmation samples has been established for this project. However, if the completeness goal is not met, EPA may still be able to make site decisions based on any or all of the remaining validated data.

1.6 Special Training/Certification Requirements:

☒ OSHA 1910
☐ Special Equipment/Instrument Operator
☐ **Other (describe below):**

Region 7 Superfund Program
Quality Assurance Project Plan Form

1.7 Documentation and Records:

☒ Field Sheets	☒ Site Log	☐ Trip Report	☒ Site Maps	☐ Video
☒ Chain of Custody	☒ Health and Safety Plan	☐ Letter Report	☒ Photos	

Sample documentation will follow EPA Region 7 SOP 2420.5.

Other: Analytical information will be handled according to procedures identified in Table 2.

2.0 Measurement and Data Acquisition:

2.1 Sampling Process Design:

☐ Random Sampling	☐ Transect Sampling	☒ Biased/Judgmental Sampling	☐ Stratified Random Sampling
☐ Search Sampling	☐ Systematic Grid	☐ Systematic Random Sampling	☐ Definitive Sampling
☐ Screening w/o Definitive Confirmation		☒ Screening w/ Definitive Confirmation	

The proposed sampling scheme for soil sampling will be biased/ judgmental, with definitive laboratory analysis, in accordance with procedures included in the Guidance for Performing Site Inspections Under CERCLA, OSWER Directive #9345.1-05, September 1992, and Removal Program Representative Sampling Guidance, Volume 1: Soil, OSWER Directive 9360.4-10, November 1991. Ten percent of samples, or approximately 60 samples, will be submitted for analysis by the EPA Region 7 laboratory. The proposed number of samples is a balance between cost and coverage and represents a reasonable attempt to meet the study objectives while staying within the budget constraints of a typical site investigation.

Sample Summary Location	Matrix	# of Samples*	Analysis
Soil screening samples	Soil	600	Total lead and zinc by XRF
Soil confirmation samples	Soil	60	Total lead and zinc

*NOTE:.

2.2 Sample Methods Requirements:

Matrix	Sampling Method	EPA SOP(s)/Methods
Soil screening samples	A grab sample will be collected using hand tools from every 6-inch interval from ground surface to a depth of 4 feet at each location. A backhoe will be used to excavate soil in 6-inch lifts. Soil will be collected from the backhoe bucket and homogenized for XRF screening in a plastic bag.	EPA SW-846, Method 6200 (XRF Field Screening)
Soil confirmation	Sufficient sample volume will be collected for the screening sample so that a confirmation sample can be placed in an 8-oz jar for analysis at the Region 7 Laboratory.	EPA Region 7 SOP 4230.19

Region 7 Superfund Program
Quality Assurance Project Plan Form

2.3 Sample Handling and Custody Requirements:

☒ Samples will be packaged and preserved in accordance with procedures defined in Region 7 EPA SOP 2420.6.
☒ CoC will be maintained as directed by Region 7 EPA SOP 2420.4.
☒ Samples will be accepted according to Region 7 EPA SOP 2420.1.
☐ **Other (Describe):**

2.4 Analytical Methods Requirements:

☐ Identified in attached table.
☐ Identified in attached Analytical Services Request (ASR) Form.
☒ **Other (Describe):** Identified in future ASR Form.

2.5 Quality Control Requirements:

☐ Not Applicable
☒ Identified in attached Table 1.
☒ In accordance with the **Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites (June 2007).**

Describe Field QC Samples to be Collected: All QC samples will be submitted for the analyses listed in Table 1 (attached). Precision and accuracy will be evaluated based on the MS/MSD and field duplicate samples, and representativeness will be evaluated with the field duplicates. Evaluation of blank samples depends on the levels of contamination found in environmental samples to determine whether the environmental samples are representative.
☐ **Other (Describe):**

2.6. Instrument/Equipment Testing, Inspection, and Maintenance Requirements :

☐ Not Applicable
☒ In accordance with the **Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites (June 2007).**
☒ **Other (Describe):** Testing, inspection, and maintenance of field instruments (GPS unit, XRF unit, etc.) will be performed in accordance with manufacturers' recommendations. Testing, inspection, and maintenance of laboratory equipment will be performed in accordance with referenced SOPs and/or manufacturers' recommendations.

2.7 Instrument Calibration and Frequency:

☐ Not Applicable
☒ Inspection/acceptance requirements are in accordance with the **Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites (June 2007)**.
☒ Calibration of laboratory equipment will be performed as described in the referenced SOPs and/or manufacturers' recommendations.
☒ **Other (Describe):** Calibration of field instruments will be performed daily as described in the manufacturers' recommendations.

2.8 Inspection/Acceptance Requirements for Supplies and Consumables:

☐ Not Applicable
☒ In accordance with the **Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites (June 2007).**
☒ All sample containers will meet EPA criteria for cleaning procedures for low-level chemical analysis. Sample containers will have Level II certifications provided by the manufacturer in accordance with precleaning criteria established by EPA in *Specifications and Guidelines for Obtaining Contaminant-Free Containers.*
☐ **Other (Describe):**

2.9 Data Acquisition Requirements:

☐ Not Applicable
☒ In accordance with the **Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites** (**June 2007).**
☒ Previous data/information pertaining to the site (including other analytical data, reports, photos, maps, etc., which are referenced in this QAPP) have been compiled by EPA and/or its contractor(s) from other sources. Some of that data has not been verified by EPA and/or its contractor(s); however, the information will not be used for decision-making purposes by EPA without verification by an independent professional qualified to verify such data/information.
☐ **Other (Describe):**

2.10 Data Management:

☒ All laboratory data acquired will be managed in accordance with Region 7 EPA SOP 2410.1.
☐ **Other (Describe):**

3.0 Assessment and Oversight:

3.1 Assessment and Response Actions:

☒ Peer Review ☒ Management Review ☐ Field Audit ☐ Lab Audit
☒ Assessment and response actions pertaining to analytical phases of the project are addressed in Region 7 EPA SOPs 2430.6 and 2430.12.
☐ **Other (Describe):**

3.1A Corrective Action:

☒ Corrective actions will be taken at the discretion of the EPA project manager, whenever there appear to be problems that could adversely affect data quality and/or resulting decisions affecting future response actions pertaining to the site.
☐ **Other (Describe):**

Region 7 Superfund Program
Quality Assurance Project Plan Form

3.2 Reports to Management:

☐ Audit Report ☐ Data Validation Report ☐ Project Status Report ☐ None required

☐ A letter report describing the sampling techniques, locations, problems encountered (with resolutions to those problems), and interpretation of analytical results will be prepared by HGL and submitted to EPA.

☒ Reports will be prepared in accordance with the **Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites** (**June 2007)**.

☒ **Other (Describe):** A Preliminary Design Report will be submitted for this task order.

4.0 Data Validation and Usability:

4.1 Data Review, Validation, and Verification Requirements:

☐ Identified in attached table.

☒ Data review and verification will be performed in accordance with the **Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites** (**June 2007).**

☒ Data review and verification will be performed by a qualified analyst and the laboratory's section manager as described in Region 7 EPA SOPs 2430.6 and 2430.12.

☐ **Other (Describe):**

4.2 Validation and Verification Methods:

☐ Identified in attached table.

☒ The data will be validated in accordance with Region 7 EPA SOPs 2430.6 and 2430.12.

☒ The EPA TOPO will inspect the data to provide a final review. EPA lab personnel will review the data, if applicable, for laboratory spikes and duplicates, laboratory blanks, and the field blank to ensure that they are acceptable. EPA lab personnel will also compare the sample descriptions with the field sheets for consistency and will ensure that any anomalies in the data are appropriately documented.

☐ **Other (Describe):**

4.3 Reconciliation with User Requirements:

☐ Identified in attached table

☒ If data quality indicators do not meet the project's requirements as outlined in this QAPP, the data may be discarded and re-sampling or re-analysis of the subject samples may be required by the EPA project manager.

☐ **Other (Describe):**

Table 1: Sample Summary

Site Name: Former Cherokee County OU8 | **City:** Cherokee County, Kansas

HGL Project Manager: Andrea Fletcher (TO73) | **Activity/ASR #:** TBD | **Date:** June 2017

Number of Samples	Matrix	Location	Purpose	Depth or other Descriptor	Requested Analysis	Sampling Method	Analytical Method/SOP
TO73							
Up to 600	Soil	Up to 60 locations based on rail line inventory	Assess potential contamination from construction materials in rail lines	Up to 8 samples at a 6-inch intervals vertically at each location from the surface to 4 feet bgs, and up to 4 samples horizontally at half the sample locations	Total lead and zinc	EPA SW-846, Method 6200 (XRF Field Screening) & SOP 4230.19	XRF
Up to 60	Soil	Co-located with select XRF locations	Confirm XRF screening results	0-6 inches collected at select locations	Total lead and zinc	SOP 4230.19	SW846 6010B/6020
QC Samples							
TO73							
Up to 6	Soil	Duplicates	QC	Variable	Total lead and zinc	SOP 4230.19	SW846 6010B/6020
Up to 6	Soil	MS/MSD	QC	Variable	Total lead and zinc	SOP 4230.19	SW846 6010B/6020

Notes:
ASR = analytical services request
bgs = below ground surface
EPA = U.S. Environmental Protection Agency
HGL = HydroGeoLogic, Inc.
MS/MSD = matrix spike/matrix spike duplicate
QC = quality control
SOP = standard operating procedure
TBD = to be determined
TO = task order
XRF = x-ray fluorescence

Table 2: Data Quality Objectives Summary								
Site Name: Former Cherokee County OU8					**City:** Cherokee County, Kansas			
HGL Project Manager: Andrea Fletcher (TO73)					**Activity/ASR #:** TBD		**Date:** May – June 2017	
Analysis	**Analytical Method**	**Data Quality Measurements**					**Sample Handling Procedures**	**Data Management Procedures**
		Accuracy	**Precision**	**Representativeness**	**Completeness**	**Comparability**		
Soil (TO 73)								
See Table 1	See Table 1	per analytical method	per analytical method	Biased/judgmental sampling based on professional judgment of the sampling team. XRF screening with confirmation sampling	10%	Standardized procedures for sample collection and analysis will be used	See Section 2.3 of the Generic QAPP	See Section 2.10 of the Generic QAPP form

Notes:
% = percent
ASR = analytical services request
HGL = HydroGeoLogic, Inc.
OU = operable unit
QAPP = Quality Assurance Project Plan
TBD = to be determined
TO = task order
XRF = x-ray fluorescence

Table 3: EPA Approved Methods					
Matrix	**Location**	**Purpose**	**Requested Analysis**	**Sampling Methods**	**Analytical Method**
Samples					
Soil	Former Rail Beds throughout Cherokee County	Confirm XRF readings obtained in the field	Total lead and zinc	EPA SOPs 4231.1707 and 4231.2012	EPA Method 3050B/6010B
QC Samples					
Soil	Field duplicates	Assess the precision of analytical and sampling methods	Total lead and zinc	EPA SOPs 4231.1707 and 4231.2012	EPA Method 3050B/6010B

Notes:
EPA = U.S. Environmental Protection Agency
QC = quality control
SOP = standard operating procedure
XRF = x-ray fluorescence

Appendix B

Sample Collection Field Sheet

Sample Collection Field Sheet

US EPA Region 7
Kansas City, KS

ASR Number: **Sample Number:** **QC Code:** **Matrix:** **Tag ID:**

Project ID No.: **EPA Project Manager:**

Project Desc:

City: **State:**

Program:

Location Desc:
_____

External Sample Number: _____

Expected Concentration (Circle One): **Low** **Medium** **High** **Date:**
Time(24hr):

Latitude: ___ ___ ___ **Sample Collection:** **Start** ___/___/___ ____:____
Longitude: ___ ___ ___ **End** ___/___/___ ____:____

Field Measurements:

Parameter	**Value**	**Units**
Conductance, Specific	_____	umhos/cm
pH	_____	SU

Laboratory Analyses:

Container	**Preservative**	**Holding**	**Time**	**Analysis Name**

Sample Comments:

Sample Collected By: _____

Appendix C

Example of a Daily Quality Control Report (DQCR)

DAILY QUALITY CONTROL REPORT

Project Manager: _____

Project: _____

Date: _____

S	M	T	W	TH	F	S

Weather	Bright Sun	Clear	Overcast	Rain	Snow
Temp	To 32	32-50	50-70	70-85	>85
Wind	Still	Moderate	High	Gusty	
Humidity	Dry	Moderate	Humid		

Personnel on Site: _____

Contractors on Site: _____

Visitors on Site: _____

Work Performed: _____

Sheet 1 of 2

Project: _____ **Date:** _____

Quality Control Activities (including field calibration and duplicate samples collected): _____

Problems Encountered/Corrective Actions Taken: _____

Downtime/Standby: _____

Health and Safety Activities: _____

Special Notes: _____

By: _____ **Date:** _____

Sheet 2 of 2

Appendix D

Example of a Chain-of-Custody (COC) Form

SAMPLE CHAIN OF CUSTODY

Project Name: _____ **Project Location:** _____

Activity Number: _____

Project Manager: _____

Samplers: _____

Sample Date	Time	Sample Identification	Preservative	No. of Containers	Type of Containers	Analysis

Remarks/Additional or Special Analyses: _____

Signatures	Date	Time	Mode of Shipment	Reason for change of custody
Relinquished By:				
Received By:				
Relinquished By:				
Received in Laboratory By:				

APPENDIX B

SITE SAFETY AND HEALTH PLAN

Site Safety and Health Plan Form	Environmental Protection Agency --Region 7--	HydroGeoLogic, Inc.
		Project Name: Cherokee County Superfund Site OU8 Railroads Task Order No.: 0073

Job Site Address: **Various relic rail lines throughout Cherokee County Site, Cherokee County, Kansas**	**Task Order Manager: Andrea Fletcher** **WBS Work Area: RD**
Site Contact: Todd Campbell	**EPA TOPO: Elizabeth Hagenmaier**
Telephone: 913-669-5479	**Telephone: 913-551-7939**
☐ **Revision No. 0**	**email: hagenmaier.elizabeth@epa.gov**
Objectives of Fieldwork: To collect soil samples to better delineate the contamination within OU8. The data gathered will be used for the remedial design (RD). Tasks that will be conducted to meet the project objectives include Site visits (with no intrusive work) to observe Site conditions and plan fieldwork; screening of soil samples using XRF, and collection of soil samples for confirmation analysis. A backhoe will be used to excavate test pits across the rail line ballasts; grab samples will be collected using hand tools from every 6-inch interval from the ground surface to a depth of 4 feet at each location. Soil will be collected from the backhoe bucket and homogenized for XRF screening in a disposable pan. The test pits will not be entered by sampling personnel. Up to 660 grab samples will be collected. Grab samples will be screened by HGL and EPA personnel using an XRF. Up to 66 grab samples will be collected for confirmation sampling purposes and will be sent to the EPA Region 7 laboratory for analysis.	**Type: Check as many as applicable** ☐ Active ☐ Landfill ☐ Unknown ☒ Inactive ☐ Uncontrolled ☐ Military ☐ Secure ☐ Industrial ☐ Enclosed space ☒ Unsecure ☐ Recovery ☐ Well Field ☐ Other **specify: Former mining area, tailings piles/pits**

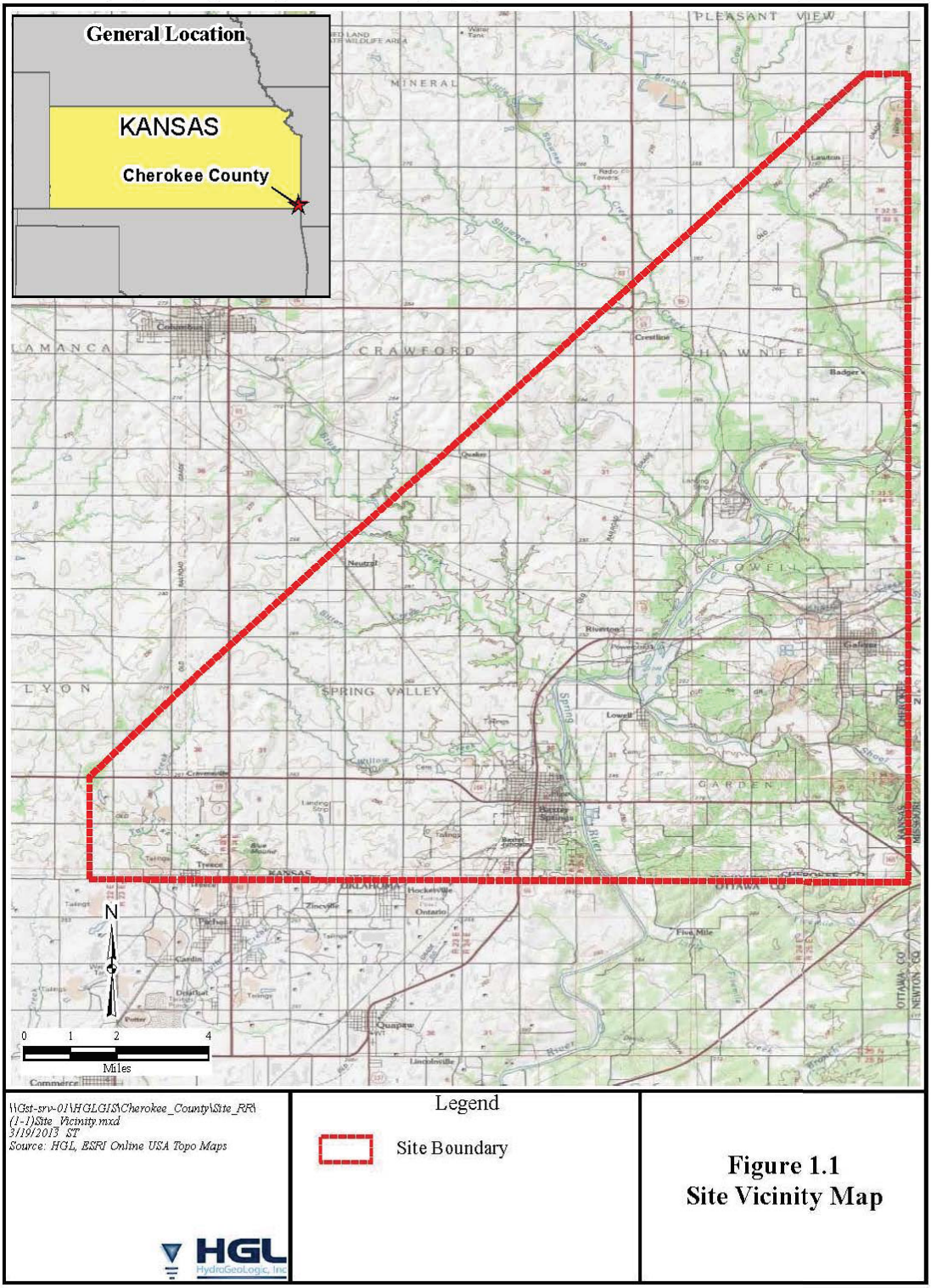
Description and Features:

Fieldwork will be conducted only along abandoned railroads within Cherokee County that were associated with the historic mining within the county. These rail lines were abandoned by railroad companies and reverted back to the property owner through the Surface Transportation Board. Because fieldwork will be conducted only along these abandoned railroads, no access for any active railroads is required and no hazards associated with active rail lines are anticipated.

The Site topography is generally gently sloping, except in the area of waste stockpiles and collapsed mine areas. Topographic relief in the stockpile areas approaches 50 feet. Topographic relief associated with existing mine shafts and collapse features is on the order of 50 to 100 feet. Side slopes along the collapse features are very steep. Fieldwork and sample collection will remain along abandoned railroad lines where the topography is expected to be gently sloping, except along the steeper slopes of the railroad embankments.

Broad areas of mining waste were observed in various areas of the Site where little or no vegetation is present. In contrast, mature woodlands are present adjacent to mine waste areas and the collapse areas. Other areas exhibit spotty, native grasses and brush with occasional groupings of trees. These native grasses, brush, and occasional groupings of trees are anticipated along the abandoned railroad lines.

Surrounding Population: ☐ Residential ☐ Industrial ☒ Rural ☐ Urban ☐ Other:



For more detailed site maps, see the figures in the Field Sampling Plan (FSP).

Site Safety and Health Plan Form	Environmental Protection Agency --Region 7--	HydroGeoLogic, Inc.
		Project Name: Cherokee County Superfund Site OU8 Railroads
		Task Order No.: 0073

Site History:
The Cherokee County Superfund Site (Site) encompasses the Kansas portion of the Tri-State Mining District in the southeast corner of the state. The Tri-State Mining District covers approximately 2,500 square miles in northeast Oklahoma, southwest Missouri and southeast Kansas. The Tri-State Mining District was one of the foremost lead-zinc mining areas of the world and provided nearly continuous production from about 1850 until 1970. During this period the district produced an estimated 500 million tons of ore, with about 115 million tons produced from the Kansas portion of the district. EPA has formerly listed four mining related Superfund Sites in the Tri-State Mining District: the Tar Creek Site in Oklahoma, the Jasper County and Newton County in Missouri, and the Cherokee County Site in Kansas.

The District is characterized by a variety of mine waste features that contain sparse to no vegetation. Local stream systems also contain mining wastes and mining-impacted sediments and surface water. Residential areas are adjacent to mine waste accumulations in some areas or have suffered historic impacts as a result of smelting. Lead and zinc are found in mining wastes and soils at maximum concentrations of several thousand parts per million (ppm).

During the mining years, railroads were constructed in Cherokee County to join conventional large-scale railroads to the individual mining operations. As of 2000, approximately 142 miles of large-scale rail lines exist in Cherokee County. Historically, the ballast used in the railroad beds was composed of chat from surrounding mine waste piles. Traditionally, these historical railroads were abandoned when mining operations ceased in that mine. Currently, the historic railroads that cross through private property exhibit extensive regrowth. The organic layer covering the chat ballast in forested areas is well developed owing to the almost constant supply of litter from the surrounding vegetation.

Recently, many rail lines were abandoned by railroad companies and reverted back to the property owner through the Surface Transportation Board. Plans also exist to convert some historic rail beds to the national Rails to Trails program. Contamination at several historical rail lines have been addressed during previous remedial actions on properties where they were encountered. Some ballast may have been completely removed during subsequent construction activities, such as highway cuts. With the potential changes in land use, the exposure scenarios have changed and the rail lines are being investigated systematically.

Waste Types: ☐ Liquid ☒ Solid ☐ Sludge ☐ Gas ☐ Unknown ☐ Other **Specify:**

Waste Characteristics: Check as many as applicable.

☐ Corrosive	☐ Flammable	☐ Radioactive*
☒ Toxic	☐ Volatile	☐ Reactive
☐ Inert Gas	☐ Unknown	☒ Carcinogenic
☐ Other **Specify:**		

*Contact CSHD for further project planning.

Work Zones:
Work zones will be used during screening. The exclusion zone will be areas in proximity to sampling areas. The support zone will be considered the 10-foot perimeter around support vehicles.

Hazards of Concern:

Indoors	**Outdoors**
☐ Exhaust	☒ Noise
☐ Inorganic Chemicals	☒ Inorganic Chemicals
☐ Organic Chemicals	☐ Organic Chemicals
☐ Motorized Traffic	☐ Motorized Traffic
☐ Heavy Machinery	☒ Heavy Machinery
☐ Slips, Trips, and Falls	☒ Slips, Trips, and Falls
☐ Noise	☒ Biological: stinging insects, venomous reptiles
☐ Explosive/Flammable	☒ Cold Stress
☐ Radiological	☒ Heat Stress
☐ Other Specify	

Principle Disposal Methods and Practices for investigation derived waste. Summarize below:

Soil IDW will be replaced into excavations. Expendable equipment and used PPE will be double bagged and disposed of as municipal waste. Dry decontamination methods will be used on excavation equipment.

Site Safety and Health Plan Form	Environmental Protection Agency --Region 7--	HydroGeoLogic, Inc.
		Project Name: Cherokee County Superfund Site OU8 Railroads
		Task Order No.: 0073

Project Specific Hazardous Material Summary: Circle waste type and media in which the material is contained, estimate quantity if material exists in bulk quantities.

Chemicals Amounts/Units:	Solids Amounts/Units:	Sludges Amounts/Units:	Solvents Amounts/Units:	Oils Amounts/Units:	Other Amounts/Units:
☐ Acids	☐ Flyash	☐ Paint	☐ Halogenated (chloro, bromo)	☐ Oily Wastes	☐ Laboratory
☐ Pickling Liquors	☐ Asbestos	☐ Pigments	☐ Solvents	☐ Gasoline	☐ Pharmaceutical
☐ Caustics	☒ Milling/Mine Tailings	☐ Metal Sludges	☐ Hydrocarbons	☐ Diesel Oil	☐ Hospital
☐ Pesticides	☐ Ferrous Smelter	☐ POTW Sludge	☐ Alcohols	☐ Lubricants	☐ Radiological
☐ Dyes/Inks	☐ Non-ferrous Smelter	☐ Aluminum	☐ Ketones	☐ PCBs	☐ Municipal
☐ Cyanides	☒ Metals: lead, zinc	☐ Distillation Bottoms	☐ Esters	☐ Polycyclic Aromatics	☐ Construction
☐ Phenols	☐ Other	☐ Other	☐ Ethers	☐ Other	☐ Munitions
☐ Halogens	**Specify:**	**Specify:**	☐ Other	**Specify:**	☐ Other
☐ Dioxins	**Site is principally a series of chat piles and tailings left after mining operations ceased. OU8 includes ballast and soil on the remaining rail lines throughout the county.**		**Specify:**		**Specify:**
☐ Other					
Specify:					

Overall Hazard Evaluation: ☐ High ☐ Medium ☒ Low ☐ Unknown (Where tasks have different hazards, evaluate each. Attach additional sheets if necessary.)

Justification: Exposure routes are through ingestion and inhalation of metals-contaminated soil; dermal contact, and/or inhalation of particulates entrained in air. Proper personal hygiene (i.e., laundering, showering, and washing hands) after field activities and the use of proper PPE (gloves) during soil screening will minimize ingestion of contaminants and take home toxic potential. If ambient dust is noted, engineering controls will be implemented, such as working upwind and the application of water for dust suppression.

Fire/Explosion Potential: ☐ High ☐ Medium ☒ Low ☐ Unknown

Background Review: ☒ Complete ☐ Incomplete Additional information to be collected in this and future investigations.

Known Contaminants (CAS Number)	Highest Observed Concentration (specify units and media)	TLV ppm or mg/m^3 (specify)	STEL/ Ceiling Limit	IDLH ppm or mg/m^3 (specify)	Symptoms/Effects of Acute Exposure	PID Potential
Lead (7439-92-1)	13,000 mg/kg in subsurface soil [1]	0.05 mg/m^3 0.03 mg/m^3 Action Level	0.05 mg/m^3	100 mg/m^3	Central nervous system impairment; lower respiratory tract impairment; hematological effects	None
Zinc (7440-66-6)	52,000 mg/kg in subsurface soil [1]	2 mg/m^3 (respirable, as zinc oxide)	10 mg/m^3 (respirable)	500 mg/m^3	Metal fume fever; chills; muscle aches; metallic taste in mouth	None

Notes:
[1] Reported in the ROD for the Baxter Springs and Treece Subsites.
ACGIH = American Conference of Governmental Industrial Hygienists
CAS = Chemical Abstract Service
IDLH = Immediately Dangerous to Life and Health (NIOSH standard enforced by law)
mg/m^3 = milligrams per cubic meter
NE = Not established
NIOSH = National Institute for Occupational Safety and Health
OSHA = Occupational Safety and Health Administration
PID = photoionization detector
ppm = parts per million
(R) = Respirable fraction
STEL = Short Term Exposure Limit (15 minute)
TLV = Threshold Limit Values (Recommended by ACGIH) over 8-hr work shift
mg/kg = milligrams per kilogram

Chemical	Exposure Limit mg/m^3	Maximum Soil Concentration mg/kg	Exposure Limit Based on Single Compound mg/m^3
Lead	**0.05**		**0.96**

Action level for total dust monitor should be 1 mg/m^3 (rounded up from 0.96 mg/m^3).

Site Safety and Health Plan Form	Environmental Protection Agency --Region 7--	HydroGeoLogic, Inc.
		Project Name: Cherokee County Superfund Site OU8 Railroads Task Order No.: 0073

Activity Hazard Analysis – Complete Corresponding Detailed PPE Section for each task

Task Number/Activity Description/ Site Location (attach additional sheets if necessary)	Potential Hazards	Controls	Primary /Secondary PPE Level	Exposure Monitoring Required/ Frequency
1 Site Visit/Field Mapping (visual inspection/observation only)	Slips, trips and falls	Awareness of surroundings	D/D	Not Required
	Biological Hazards	Appropriate outerwear, awareness of surroundings, insect/tick repellant, poison ivy barrier creams, shower upon leaving work		
	Cold/Heat stress	Schedule breaks according to weather, appropriate PPE		
	UV exposure	Sunscreen		
2 Sample Collection - grab samples will be collected using hand tools from every 6-inch interval from ground surface to a depth of 4 feet at each location. A backhoe will be used to excavate soil in 6-inch lifts. Soil will be collected from the backhoe bucket and homogenized for screening in a disposable pan. The test pits will not be entered by sampling personnel. HGL and EPA personnel will scan the soil samples using the XRF. Soil samples will be collected at a frequency of 10 percent for confirmation analysis.	Traffic	High Visibility Vests, Traffic cones	D/D	Action level: 1 mg/m^3 Dust suppression will be use to keep airborne concentrations below 1 mg/m^3
	Machinery	Locate support equipment away from mechanical activities. Signal; make eye contact w/ operator to stop operation before entering excavator swing radius. Keep hands and feet, loose clothing, other articles away from rotating equipment. Use a spotter. Keep hands and feet clear when moving equipment.		
	Chemical exposure	Appropriate PPE. Dust will be controlled using engineering controls—dust suppression by the application of water and working upwind of the excavation. Continuous dust monitoring during test pit work.		
	Cold/Heat stress	Schedule breaks according to weather, appropriate PPE, sunscreen		
	Noise	Hearing Protection as required		
	Biological Hazards	Appropriate outerwear, awareness of surroundings, insect/tick repellant, poison ivy barrier creams, shower upon leaving work		
	Slips, trips and falls	Good housekeeping practices, be alert, no entry into test pits unless active digging has stopped and pit is less than 3 feet deep.		
	UV exposure	Sunscreen		

PPE Levels = A, B, C, D – definitions of these levels available in Corporate H&S Manual.
Exposure Monitoring = PDR (dust monitor)

Site Safety and Health Plan Form | **Environmental Protection Agency --Region 7--** | **HydroGeoLogic, Inc.**

Project Name: Cherokee County Superfund Site OU8 Railroads
Task Order No.: 0073

Does the project require continuous air monitoring during the project?
☒ Yes ☐ No Ambient dust monitoring during intrusive activities that could generate dust. Monitor type: MIE PDR-1000

Protective Equipment: Specify by task. Indicate type and/or material as necessary. Use copies of this sheet if needed.

Task Number: 1 and 2 ☒ **Primary**
PPE Level: Modified D ☒ **Contingency**

Protective Clothing: ☒ Not Needed
- ☐ Encapsulated Suit:
- ☐ Splash Suit:
- ☐ Apron
- ☐ High Visibility Vests:
- ☐ Coverall Specify:

Respiratory: ☒ Not Needed
- ☐ SCBA, Airline:
- ☐ APR Full face:
- ☐ Cartridge:
- ☐ Escape Mask:
- ☐ Other:

Gloves: ☐ Not Needed
- ☐ Undergloves
- ☐ Overgloves
- ☒ Gloves: Specify: Nitrile (6 mil) When handling potentially contaminated soils.

Boots: ☐ Not Needed
- ☒ Boots: Leather steel-toe
- ☐ Overboots:
- ☐ Rubber:

Head & Eye: ☐ Not Needed
- ☒ Safety Glasses: When flying debris/dust exists
- ☐ Face Shield:
- ☐ Goggles
- ☒ Hard Hat: When overhead dangers exist
- ☒ Hearing Protection: When near excavation equipment
- ☐ Other:

☒ Other – specify below: Outerwear appropriate for weather conditions.

Task Number: ☐ **Primary**
PPE Level: ☐ **Contingency**

Protective Clothing: ☐ Not Needed
- ☐ Encapsulated Suit:
- ☐ Splash Suit:
- ☐ Apron
- ☐ High Visibility Vests:
- ☐ Coverall Specify:

Respiratory: ☐ Not Needed
- ☐ SCBA, Airline:
- ☐ APR Full face:
- ☐ Cartridge:
- ☐ Escape Mask:
- ☐ Other:

Gloves: ☐ Not Needed
- ☐ Undergloves
- ☐ Overgloves
- ☐ Gloves: Specify: Nitrile (6 mil) When there is the potential for contact with potentially contaminated surface water.

Boots: ☐ Not Needed
- ☐ Boots: Leather steel-toe
- ☐ Overboots:
- ☐ Rubber:

Head & Eye: ☐ Not Needed
- ☐ Safety Glasses: Where splash potential exists
- ☐ Face Shield:
- ☐ Goggles
- ☐ Hard Hat:
- ☐ Hearing Protection:
- ☐ Other:

☐ Other – specify below: Outerwear appropriate for weather conditions.

Task Number: ☐ **Primary**
PPE Level: ☐ **Contingency**

Protective Clothing: ☐ Not Needed
- ☐ Encapsulated Suit:
- ☐ Splash Suit:
- ☐ Apron
- ☐ High Visibility Vests:
- ☐ Coverall Specify:

Respiratory: ☐ Not Needed
- ☐ SCBA, Airline:
- ☐ APR Full face:
- ☐ Cartridge:
- ☐ Escape Mask:
- ☐ Other:

Gloves: ☐ Not Needed
- ☐ Undergloves
- ☐ Overgloves
- ☐ Gloves: Specify: Nitrile (6 mil)

Boots: ☐ Not Needed
- ☐ Boots: Leather steel-toe
- ☐ Overboots:
- ☐ Rubber:

Head & Eye: ☐ Not Needed
- ☐ Safety Glasses:
- ☐ Face Shield:
- ☐ Goggles
- ☐ Hard Hat:
- ☐ Hearing Protection:
- ☐ Other:

☐ Other – specify below:

Task Number: ☐ **Primary**
PPE Level: ☐ **Contingency**

Protective Clothing: ☐ Not Needed
- ☐ Encapsulated Suit:
- ☐ Splash Suit:
- ☐ Apron
- ☐ High Visibility Vests:
- ☐ Coverall Specify:

Respiratory: ☐ Not Needed
- ☐ SCBA, Airline:
- ☐ APR Full face:
- ☐ Cartridge:
- ☐ Escape Mask:
- ☐ Other:

Gloves: ☐ Not Needed
- ☐ Undergloves
- ☐ Overgloves
- ☐ Gloves: Specify: Nitrile (6 mil)

Boots: ☐ Not Needed
- ☐ Boots: Leather steel-toe
- ☐ Overboots:
- ☐ Rubber:

Head & Eye: ☐ Not Needed
- ☐ Safety Glasses:
- ☐ Face Shield:
- ☐ Goggles
- ☐ Hard Hat:
- ☐ Hearing Protection:
- ☐ Other:

☐ Other – specify below:

Site Safety and Health Plan Form	Environmental Protection Agency --Region 7--	HydroGeoLogic, Inc.
		Project Name: Cherokee County Superfund Site OU8 Railroads Task Order No.: 0073

Personnel and Responsibilities *(Include subcontractors)*

Name	Firm/Region	Medical Monitoring Clearance* (yes/no)	Responsibilities	On-site Involvement
Andrea Fletcher	HGL/KC		TOM	All
Beatty Hean	HGL/KC	Yes	Field Screening and Oversight	All
HGL Personnel	HGL/KC	Yes	Field Screening and Oversight	All
Survey Subcontractor	TBD		Surveying	Task 1
Excavation Subcontractor	TBD		Test Pit Excavation	Task 2

*Health clearance meets all the medical surveillance requirements of 29 CFR 1910.120. Medical surveillance certification for on-site personnel is presented in HGL Policy 3.1. Subcontractors are required to meet the medical requirements of 29 CFR 1910.120, if applicable

Does the project have any permit required confined spaces that will need to be entered to accomplish the identified project tasks?
☐ Yes ☒ No If Yes, Attach the Permit Required Confined Space Entry Checklist (available via the Intranet) and contact the CHSD for additional project safety planning.

Health and Safety Monitoring Equipment: Specify by task. Indicate type as necessary. Attach additional sheets as necessary.

Instrument	Task	Action Guidelines	Comments (Include schedules of use)
Combustible Gas Indicator LEL/O_2 Meter			☒ Not Needed
Photoionization Detector Type: Multi-Rae			☒ Not Needed
Sound level Meter Type:			☒ Not Needed
Dust Monitor (Digital) Type: MIE PDR-1000	2	Engineering controls will be implemented to minimize exposure to dust, including remaining upwind of the excavation work area and applying water to the work area for dust suppression. **Action level for total dust monitor is 1 mg/m^3.**	☐ Not Needed

Decontamination Procedures

Personalized Decontamination

Wash well before hand-to-mouth contact is made. A shower will be taken as soon as possible after leaving the field. Workers will remove protective clothing in this order:

Wet or dry decontamination procedures will be selected per project.

Dry Decon Procedure
Place all disposable PPE in a garbage bag as removed in the following order:
(1) Brush off work boots, remove disposable over boots, or booties
(2) remove gloves
(3) remove safety glasses
(4) remove Tyvek or cloth coverall, if used
(5) remove respirator, if used
(6) remove inner gloves
(7) wash hands/face before eating/drinking

Wet Decon Procedure ☒ **Not Needed**
(1) wash overboots in soapy water and rinse
(2) remove overboots or booties
(3) remove gloves
(4) remove safety glasses
(5) remove Tyvek or cloth coverall, if used
(6) remove respirator, if used
(7) remove inner gloves
(8) wash hands/face before eating/drinking

Sampling Equipment Decontamination

All sampling equipment will be thoroughly decontaminated as follows:

(1) wash and scrub with low phosphate detergent
(2) potable tap water rinse 1
(3) potable tap water rinse 2
(4) thoroughly rinse with deionized water, if specified by the Work plan
(5) thoroughly rinse with solvent (e.g., hexane and/or methanol), if specified by the Work plan
(6) air dry
(7) wrap in aluminum foil for transport, if specified by the Work Plan

☒ Not Needed

Heavy Equipment Decontamination

Prior to removal from the work site, potential contaminated soil/groundwater will be scraped or brushed from the exterior surfaces.

☐ Not Needed

Containment and Disposal Method

All disposable PPE will be double-bagged prior to disposal.

Decon water to be disposed on site, if needed.

☐ Not Needed

Containment and Disposal Method

☒ Not Needed

Containment and Disposal Method

All disposable PPE will be double-bagged before disposal.

Decon water to be disposed on site, if needed.

☐ Not Needed

Hazardous Materials Inventory (MSDSs) for Investigation-Associated Substances will be available on site in the HAZCOM Binder. No Investigation-Associated Substances are anticipated for this project.

Spill Response: The following materials will be kept on site for spill response (check all appropriate materials)

☐ Absorbent Pads ☐ Granular absorbent material (nonflammable) ☒ Polyethylene Sheeting ☐ Waste Container ☒ Shovels or assorted hand tools

If a hazardous waste spill or material release to the air, soil, or water at the Site is observed, the EPA site representative and the local Fire Department will be immediately notified. An assessment will be made of the magnitude and potential impact of the release. If it is safe to do so, Site personnel will attempt to locate the source of the release, prevent further release, and contain the spilled and/or affected materials.

Site Safety and Health Plan Form	Environmental Protection Agency --Region 7--	HydroGeoLogic, Inc.
	Project Name: Cherokee County Superfund Site OU8 Railroads	
	Task Order No.: 0073	

Emergency Contacts		Emergency Contacts	Name	Phone
Emergency	911	EPA TOPO	Elizabeth Hagenmaier	913-551-7939
Dig/Utility Clearance	811	Task Order Manager/ SSHO	Andrea Fletcher	913-317-8860 W 913-488-0024 Cell
Health Department	(620) 429-3087 hd@cherokeecountyks.org	HGL Regional HSC	Phyllis Chase	913-317-8860 W 913-980-1863 Cell
Sheriff's Department	(620) 429-3992 or (620) 848-3000	HGL Corporate Health and Safety Director	Steve Davis, CSP, CIH	865-659-0499 Cell
Fire Department	Baxter Springs Fire Dept. (620) 856-3536	Occupational Physician	WorkCare	1-888-449-7787
Police Department	Baxter Springs, KS – (620) 856-2112			
Highway Patrol/State Police	(620) 431-2100 – Chanute, KS			
Poison Control Center	(417) 625-2305	**HGL 24 Hour Emergency Number**		**1-800 341-3647**
State Spill Line	1-785-291-3333			

Contingency Plans Summarize below:

If staff observes hazards for which they have not been prepared, they will withdraw from the area and call HGL CHSD Steve Davis or their regional health and safety coordinator. In the event of medical emergency, contact Hospital, Police, or Sheriff's Department.
If dust concentrations are measured that are above the action level, additional engineering controls will be implemented. If these controls do not eliminate the exposure, personnel protection will be reevaluated.
The weather will be monitored routinely. If lightning is seen or thunder heard, the "30-30 Rule" shall be used.
In the case that immediate shelter is required all personnel will go to the nearest available shelter and wait until hazardous conditions pass.

Health and Safety Plan Approvals

Prepared by: P Chase	Date: 03/28/17
SSHO Signature:	Date:
HGL CSHD Signature: Edith Scala-Hampson	Date: Digitally signed by Edith Scala-Hampson Date: 2017.03.29 14:38:35 -05'00'
Site: Cherokee County Superfund Site OU8 Railroads	

Medical Emergency

Hospital Name:

Maude Norton Memorial Hospital

Hospital Address:
220 N. Pennsylvania Ave.
Columbus, KS 66725

Phone Number:

(620) 429-2545

Name of Contact at Hospital: NA

Name of 24-Hour Ambulance: NA

Route to Hospital (See Figure 2)
Nearest hospital is Maude North Memorial Hospital in Columbus, KS
From Baxter Springs, take Hwy 166/400 West to Hwy 69 North, Exit at Maple, and head west, turn right (north) at Pennsylvania.

From Treece, Take Hwy 69 North, Exit at Maple, and head west, turn right (north) at Pennsylvania.

Distance to Hospital: 15 miles

This Page Reserved for Hospital Route Map

From Baxter Springs, take Hwy 166/400 West to Hwy 69 North, Exit at Maple, and head west, turn right (north) at Pennsylvania. Drive time is approximately 20 minutes.

From Treece, Take Hwy 69 North, Exit at Maple, and head west, turn right (north) at Pennsylvania. Drive time is approximately 20 minutes.

Site Safety and Health Plan Form	Environmental Protection Agency --Region 7--	HydroGeoLogic, Inc.
		Project Name: Cherokee County Superfund Site OU8 Railroads Task Order No.: 0073

The following personnel have read and fully understand the contents of this Health and Safety Plan and further agree to all requirements contained herein.

Name and Responsibility	Affiliation	Date	Signature

APPENDIX C

FIELD FORMS

Property Log
EPA Chain of Custody
EPA Sample Collection Field Sheet
Cherokee County Railroads Access Agreement

SAMPLE COLLECTION FIELD SHEET

U.S. Environmental Protection Agency Region VII
Kansas City, Kansas

ASR Number: **Sample Number:** ____ **QC Code:** ____ **Matrix:** **TAG ID:** ____ - ___ - ___

Activity Number: **Activity Leader:**

Activity Desc.:

Location: **State:** **Type:**

Superfund Name: **Site ID:** **Site OU:**

Location Desc.:

External Sample Number:

Expected Concentration: Circle One: Low Medium High **Date** **Time (24 Hr)**

Latitude: ____° ____N ____O **Sample Collection:** **Start** ___/___/___ ___:___

Longitude: ____° ____N ____O **End** ___/___/___ ___:___

Laboratory Analyses:

Container	Preservative	Holding Time	Analysis

Sample Comments:

Temperature (°C):_____

pH (SU):_____

Specific Conductivity (µs/cm): _____

ORP (mV): _____

D.O. (mg/L): _____

Turbidity (NTU):_____

Sample Collected By: _____

CHAIN OF CUSTODY RECORD
ENVIRONMENTAL PROTECTION AGENCY REGION VII

ACTIVITY LEADER(Print)	NAME OF SURVEY OR ACTIVITY	DATE OF COLLECTION DAY MONTH YEAR	SHEET of

CONTENTS OF SHIPMENT

SAMPLE NUMBER	TYPE OF CONTAINERS					SAMPLED MEDIA					RECEIVING LABORATORY REMARKS/OTHER INFORMATION (condition of samples upon receipt, other sample numbers, etc.)
	CUBITAINER	BOTTLE	BOTTLE	BOTTLE	VOA SET (2 VIALS EA)	water	soil	sediment	dust	other	
	NUMBERS OF CONTAINERS PER SAMPLE NUMBER										

DESCRIPTION OF SHIPMENT	MODE OF SHIPMENT
_____ PIECE(S) CONSISTING OF _____ BOX(ES) _____ ICE CHEST(S): OTHER _____	_____ COMMERCIAL CARRIER: _____ _____ COURIER _____ SAMPLER CONVEYED _____ (SHIPPING DOCUMENT NUMBER)

PERSONNEL CUSTODY RECORD

RELINQUISHED BY (SAMPLER)	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	

7-EPA-9262(Revised 5/85)

Property Log

Property # _____ **Property Address** _____

Owner's Name _____

Date/Time	Spoke To	HGL	Comments
/			
/			
/			
/			
/			
/			
/			
/			
/			

Consent for Access to Property

RIGHT OF ENTRY TO PREMISES

Grantor (the property owner), consents to and authorizes the United States Environmental Protection Agency (USEPA) or its authorized representatives, to enter and perform environmental response activities upon the following described premises:

Owner's Name	___(name)___ ___(address)___ ___(address)___
Addresses/Description of properties covered by this Agreement	Property in Sec __, T __, R ___ Parcel _____
Daytime Phone	

Grantor understands that this grant does not limit EPA's right of access under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), 42 USC. § 9601-9675, or any other law.

PURPOSE OF ACCESS

The purpose of the response activities is to remediate existing mining waste materials on the referenced property. Remediation may include excavation of the mining waste for disposal in existing pits/ponds, excavation and off-site disposal of waste materials, regrading of the waste and construction of earthen cover systems, regrading and revegetation of areas disturbed by the remedial activities, and construction of temporary and permanent storm water management structures. The initial response actions will involve collecting field information for use during design of the remedial systems. The initial field activities may include excavation of test pits to document the location, thickness and characteristics of mining wastes on the referenced property. Field testing of soil samples from the test pit excavations may be performed to evaluate metals contamination. Additionally, temporary survey stakes/monuments may be installed to establish ground control for aerial photography and development of topographic information. Once the remedial design is completed, the construction phase will begin when funding is available. During this phase, earthmoving equipment will be mobilized to the referenced property to remediate the mining waste materials. The response activities during the construction phase are summarized below.

ENVIRONMENTAL RESPONSE ACTIONS

The environmental response actions to be performed on the referenced property may include the following activities:

1. Excavating and backfilling test pits using a backhoe;
2. Drilling and backfilling soil borings using a drill rig.
3. Installing PVC piezometers to measure water levels in waste materials and underlying soil.
4. Field testing of soil samples for metals contamination;
5. Obtaining samples from Grantor's property;
6. Installing temporary survey stakes/monuments for ground control in preparation for aerial photography/surveying of the property.
7. Mobilizing earthmoving equipment to perform excavation and grading activities.
8. Field staking of areas to be remediated.
9. Excavation of mining waste for placement in on-site or off-site disposal areas.
10. Regrading of mining wastes and placement of earthen cover systems.
11. Construction of stormwater management and erosion control structures.
12. Revegetation of areas disturbed by the remedial construction activities.

TERM

This access agreement shall be operable for the period of time it takes to complete the environmental response activities. Upon completion of the response actions, all rights and privileges given by the Grantor shall cease on that date, unless extended by subsequent agreement.

AGREEMENT NOT TO INTERFERE

Grantor agrees not to interfere with any of the activities undertaken by Grantees at the Property, tamper with any of Grantees' property, or take any actions regarding the use of the Property which will endanger human health or welfare or the environment, or allow others to use the Property in such a manner during the term of this consent. Grantor agrees to provide notice and a copy of this agreement to prospective purchasers, lessees, assigns, or grantees of the property or any portion of it. Grantor agrees to provide 30-day notice to EPA prior to any transfer of ownership rights to the property.

SAMPLING RESULTS

Grantees agree to provide Grantor with the results of any and all sampling and/or analysis resulting from Grantees' response activities on the property.

PRIOR CONDITIONS AGREEMENT

The Grantor agrees to the condition of the property prior to start of the initial response actions including excavating test pits and testing of waste materials and soils.

RESTORATION OF PROPERTY

The Grantees agree to take reasonable precautions to minimize disturbance of existing vegetation to obtain access to the test pit locations. Test pits shall be promptly backfilled with the excavated material materials when field testing at the individual test pit location is completed. Backfill will be placed flush with the ground surface to restore the ground surface to its pre-excavation elevation. No seeding or sodding of areas disturbed by the field activities is planned. Field stakes/lath/monuments may be used to mark the test pits and ground control locations for future reference. During the implementation of the remedial design, Grantor understands that areas of mining waste will be removed or covered, resulting in significant land disturbance. The Grantees will take reasonable precautions to manage stormwater and erosion during the construction activities. When construction is completed, the Grantees will undertake reasonable efforts to regrade the disturbed areas to provide positive surface drainage and to revegetate the areas affected by the construction activities.

I have read the foregoing document and understand that it is an agreement granting permission to the Grantees to enter the above described premises for the purpose of remediating existing mining waste, and I agree to its terms and conditions.

_____ _____

Signature (Grantor's) **Date**

APPENDIX D

STANDARD OPERATING PROCEDURES

EPA SOP 4231.2006, *Sampling Equipment Decontamination*
EPA Region 7 SOP 2420.4C, *Field Chain of Custody for Environmental Samples*
HGL SOP 4.07, *Field Logbook Use and Maintenance*
EPA SOP 4220.03A, *Protocols for the Region 7 Lead-Contaminated Residential Yard Soil Cleanup Actions, Procedures and Sequencing*
EPA SOP 4230.19B, *Soil Sampling at Lead-Contaminated Residential Sites*
EPA SOP 2420.05D, *Identification, Documentation, and Tracking of Samples*
EPA SW-846, Method 6200, *Field Portable X-ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment*
EPA SOP 2420.6E, *Sample Container Selection, Preservation, and Holding Times*
EPA SOP 2420.1E, *Sample Receipt and Log-In*
EPA SOP 2430.6C, *Periodic Internal Program Review of the Region 7 Laboratory*
EPA SOP 2430.12E, *Regional Laboratory Quality Control Program*
EPA SOP 2410.1E, *Analytical Data Management Procedures*

SAMPLING EQUIPMENT DECONTAMINATION

SOP#: 2006
DATE: 08/11/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to provide a description of the methods used for preventing, minimizing, or limiting cross-contamination of samples due to inappropriate or inadequate equipment decontamination and to provide general guidelines for developing decontamination procedures for sampling equipment to be used during hazardous waste operations as per 29 Code of Federal Regulations (CFR) 1910.120. This SOP does not address personnel decontamination.

These are standard (i.e. typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitation, or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Removing or neutralizing contaminants from equipment minimizes the likelihood of sample cross contamination, reduces or eliminates transfer of contaminants to clean areas, and prevents the mixing of incompatible substances.

Gross contamination can be removed by physical decontamination procedures. These abrasive and non-abrasive methods include the use of brushes, air and wet blasting, and high and low pressure water cleaning.

The first step, a soap and water wash, removes all visible particulate matter and residual oils and grease. This may be preceded by a steam or high pressure water wash to facilitate residuals removal. The second step involves a tap water rinse and a distilled/deionized water rinse to remove the detergent. An acid rinse provides a low pH media for trace metals removal and is included in the decontamination process if metal samples are to be collected. It is followed by another distilled/deionized water rinse. If sample analysis does not include metals, the acid rinse step can be omitted. Next, a high purity solvent rinse is performed for trace organics removal if organics are a concern at the site. Typical solvents used for removal of organic contaminants include acetone, hexane, or water. Acetone is typically chosen because it is an excellent solvent, miscible in water, and not a target analyte on the Priority Pollutant List. If acetone is known to be a contaminant of concern at a given site or if Target Compound List analysis (which includes acetone) is to be performed, another solvent may be substituted. The solvent must be allowed to evaporate completely and then a final distilled/deionized water rinse is performed. This rinse removes any residual traces of the solvent.

The decontamination procedure described above may be summarized as follows:

1. Physical removal
2. Non-phosphate detergent wash
3. Tap water rinse
4. Distilled/deionized water rinse
5. 10% nitric acid rinse
6. Distilled/deionized water rinse
7. Solvent rinse (pesticide grade)
8. Air dry
9. Distilled/deionized water rinse

If a particular contaminant fraction is not present at the site, the nine (9) step decontamination procedure specified above may be modified for site specificity. For example, the nitric acid rinse may be eliminated if metals are not of concern at a site. Similarly, the solvent rinse may be eliminated if organics are not of

concern at a site. Modifications to the standard procedure should be documented in the site specific work plan or subsequent report.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The amount of sample to be collected and the proper sample container type (i.e., glass, plastic), chemical preservation, and storage requirements are dependent on the matrix being sampled and the parameter(s) of interest.

More specifically, sample collection and analysis of decontamination waste may be required before beginning proper disposal of decontamination liquids and solids generated at a site. This should be determined prior to initiation of site activities.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

- The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment provided that it has been verified by laboratory analysis to be analyte free (specifically for the contaminants of concern).

- The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal or industrial water treatment system.

- If acids or solvents are utilized in decontamination they raise health and safety, and waste disposal concerns.

- Damage can be incurred by acid and solvent washing of complex and sophisticated sampling equipment.

5.0 EQUIPMENT/APPARATUS

Decontamination equipment, materials, and supplies are generally selected based on availability. Other considerations include the ease of decontaminating or disposing of the equipment. Most equipment and supplies can be easily procured. For example, soft-bristle scrub brushes or long-handled bottle brushes can be used to remove contaminants. Large galvanized wash tubs, stock tanks, or buckets can hold wash and rinse solutions. Children's wading pools can also be used. Large plastic garbage cans or other similar containers lined with plastic bags can help segregate contaminated equipment. Contaminated liquid can be stored temporarily in metal or plastic cans or drums.

The following standard materials and equipment are recommended for decontamination activities:

5.1 Decontamination Solutions

- Non-phosphate detergent
- Selected solvents (acetone, hexane, nitric acid, etc.)
- Tap water
- Distilled or deionized water

5.2 Decontamination Tools/Supplies

- Long and short handled brushes
- Bottle brushes
- Drop cloth/plastic sheeting
- Paper towels
- Plastic or galvanized tubs or buckets
- Pressurized sprayers (H_2O)
- Solvent sprayers
- Aluminum foil

5.3 Health and Safety Equipment

Appropriate personal protective equipment (i.e., safety glasses or splash shield, appropriate gloves, aprons or coveralls, respirator, emergency eye wash)

5.4 Waste Disposal

- Trash bags
- Trash containers
- 55-gallon drums
- Metal/plastic buckets/containers for storage and disposal of decontamination solutions

6.0 REAGENTS

There are no reagents used in this procedure aside from the actual decontamination solutions. Table 1 (Appendix A) lists solvent rinses which may be required for elimination of particular chemicals. In

general, the following solvents are typically utilized for decontamination purposes:

- 10% nitric acid is typically used for inorganic compounds such as metals. An acid rinse may not be required if inorganics are not a contaminant of concern.
- Acetone (pesticide grade)[(1)]
- Hexane (pesticide grade)[(1)]
- Methanol[(1)]

[(1)] - Only if sample is to be analyzed for organics.

7.0 PROCEDURES

As part of the health and safety plan, a decontamination plan should be developed and reviewed. The decontamination line should be set up before any personnel or equipment enter the areas of potential exposure. The equipment decontamination plan should include:

- The number, location, and layout of decontamination stations.
- Decontamination equipment needed.
- Appropriate decontamination methods.
- Methods for disposal of contaminated clothing, equipment, and solutions.
- Procedures can be established to minimize the potential for contamination. This may include: (1) work practices that minimize contact with potential contaminants; (2) using remote sampling techniques; (3) covering monitoring and sampling equipment with plastic, aluminum foil, or other protective material; (4) watering down dusty areas; (5) avoiding laying down equipment in areas of obvious contamination; and (6) use of disposable sampling equipment.

7.1 Decontamination Methods

All samples and equipment leaving the contaminated area of a site must be decontaminated to remove any contamination that may have adhered to equipment. Various decontamination methods will remove contaminants by: (1) flushing or other physical action, or (2) chemical complexing to inactivate contaminants by neutralization, chemical reaction, disinfection, or sterilization.

Physical decontamination techniques can be grouped into two categories: abrasive methods and non-abrasive methods, as follows:

7.1.1 Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing and wearing away the top layer of the surface containing the contaminant. The mechanical abrasive cleaning methods are most commonly used at hazardous waste sites. The following abrasive methods are available:

Mechanical

Mechanical methods of decontamination include using metal or nylon brushes. The amount and type of contaminants removed will vary with the hardness of bristles, length of time brushed, degree of brush contact, degree of contamination, nature of the surface being cleaned, and degree of contaminant adherence to the surface.

Air Blasting

Air blasting equipment uses compressed air to force abrasive material through a nozzle at high velocities. The distance between nozzle and surface cleaned, air pressure, time of application, and angle at which the abrasive strikes the surface will dictate cleaning efficiency. Disadvantages of this method are the inability to control the amount of material removed and the large amount of waste generated.

Wet Blasting

Wet blast cleaning involves use of a suspended fine abrasive. The abrasive/water mixture is delivered by compressed air to the contaminated area. By using a very fine abrasive, the amount of materials removed can be carefully controlled.

7.1.2 Non-Abrasive Cleaning Methods

Non-abrasive cleaning methods work by forcing the contaminant off a surface with pressure. In general, the equipment surface is not removed using non-abrasive methods.

Low-Pressure Water

This method consists of a container which is filled with water. The user pumps air out of the container to create a vacuum. A slender nozzle and hose allow the user to spray in hard-to-reach places.

High-Pressure Water

This method consists of a high-pressure pump, an operator controlled directional nozzle, and a high-pressure hose. Operating pressure usually ranges from 340 to 680 atmospheres (atm) and flow rates usually range from 20 to 140 liters per minute.

Ultra-High-Pressure Water

This system produces a water jet that is pressured from 1,000 to 4,000 atmospheres. This ultra-high-pressure spray can remove tightly-adhered surface films. The water velocity ranges from 500 meters/second (m/s) (1,000 atm) to 900 m/s (4,000 atm). Additives can be used to enhance the cleaning action.

Rinsing

Contaminants are removed by rinsing through dilution, physical attraction, and solubilization.

Damp Cloth Removal

In some instances, due to sensitive, non-waterproof equipment or due to the unlikelihood of equipment being contaminated, it is not necessary to conduct an extensive decontamination procedure. For example, air sampling pumps hooked on a fence, placed on a drum, or wrapped in plastic bags are not likely to become heavily contaminated. A damp cloth should be used to wipe off contaminants which may have adhered to equipment through airborne contaminants or from surfaces upon which the equipment was set.

Disinfection/Sterilization

Disinfectants are a practical means of inactivating infectious agents. Unfortunately, standard sterilization methods are impractical for large equipment. This method of decontamination is typically performed off-site.

7.2 Field Sampling Equipment Decontamination Procedures

The decontamination line is setup so that the first station is used to clean the most contaminated item. It progresses to the last station where the least contaminated item is cleaned. The spread of contaminants is further reduced by separating each decontamination station by a minimum of three (3) feet. Ideally, the contamination should decrease as the equipment progresses from one station to another farther along in the line.

A site is typically divided up into the following boundaries: Hot Zone or Exclusion Zone (EZ), the Contamination Reduction Zone (CRZ), and the Support or Safe Zone (SZ). The decontamination line should be setup in the Contamination Reduction Corridor (CRC) which is in the CRZ. Figure 1 (Appendix B) shows a typical contaminant reduction zone layout. The CRC controls access into and out of the exclusion zone and confines decontamination activities to a limited area. The CRC boundaries should be conspicuously marked. The far end is the hotline, the boundary between the exclusion zone and the contamination reduction zone. The size of the decontamination corridor depends on the number of stations in the decontamination process, overall dimensions of the work zones, and amount of space available at the site. Whenever possible, it should be a straight line.

Anyone in the CRC should be wearing the level of protection designated for the decontamination crew. Another corridor may be required for the entry and exit of heavy equipment. Sampling and monitoring equipment and sampling supplies are all maintained outside of the CRC. Personnel don their equipment away from the CRC and enter the exclusion zone through a separate access control point at the hotline. One person (or more) dedicated to decontaminating equipment is recommended.

7.2.1 Decontamination Setup

Starting with the most contaminated station, the decontamination setup should be as follows:

Station 1: Segregate Equipment Drop

Place plastic sheeting on the ground (Figure 2, Appendix B). Size will depend on amount of

equipment to be decontaminated. Provide containers lined with plastic if equipment is to be segregated. Segregation may be required if sensitive equipment or mildly contaminated equipment is used at the same time as equipment which is likely to be heavily contaminated.

Station 2: Physical Removal With A High-Pressure Washer (Optional)

As indicated in 7.1.2, a high-pressure wash may be required for compounds which are difficult to remove by washing with brushes. The elevated temperature of the water from the high-pressure washers is excellent at removing greasy/oily compounds. High pressure washers require water and electricity.

A decontamination pad may be required for the high-pressure wash area. An example of a wash pad may consist of an approximately 1 1/2 foot-deep basin lined with plastic sheeting and sloped to a sump at one corner. A layer of sand can be placed over the plastic and the basin is filled with gravel or shell. The sump is also lined with visqueen and a barrel is placed in the hole to prevent collapse. A sump pump is used to remove the water from the sump for transfer into a drum.

Typically heavy machinery is decontaminated at the end of the day unless site sampling requires that the machinery be decontaminated frequently. A separate decontamination pad may be required for heavy equipment.

Station 3: Physical Removal With Brushes And A Wash Basin

Prior to setting up Station 3, place plastic sheeting on the ground to cover areas under Station 3 through Station 10.
Fill a wash basin, a large bucket, or child's swimming pool with non-phosphate detergent and tap water. Several bottle and bristle brushes to physically remove contamination should be dedicated to this station . Approximately 10 - 50 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.

Station 4: Water Basin

Fill a wash basin, a large bucket, or child's swimming pool with tap water. Several bottle and bristle brushes should be dedicated to this station. Approximately 10-50 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.

Station 5: Low-Pressure Sprayers

Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to contain the water during the rinsing process. Approximately 10-20 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.

Station 6: Nitric Acid Sprayers

Fill a spray bottle with 10% nitric acid. An acid rinse may not be required if inorganics are not a contaminant of concern. The amount of acid will depend on the amount of equipment to be decontaminated. Provide a 5-gallon bucket or basin to collect acid during the rinsing process.

Station 7: Low-Pressure Sprayers

Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to collect water during the rinsate process.

Station 8: Organic Solvent Sprayers

Fill a spray bottle with an organic solvent. After each solvent rinse, the equipment should be rinsed with distilled/deionized water and air dried. Amount of solvent will depend on the amount of equipment to decontaminate. Provide a 5-gallon bucket or basin to collect the solvent during the rinsing process.

Solvent rinses may not be required unless organics are a contaminant of concern, and may be eliminated from the station sequence.

Station 9: Low-Pressure Sprayers

Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to collect water during the rinsate process.

Station 10: Clean Equipment Drop

Lay a clean piece of plastic sheeting over the bottom

plastic layer. This will allow easy removal of the plastic in the event that it becomes dirty. Provide aluminum foil, plastic, or other protective material to wrap clean equipment.

7.2.2 Decontamination Procedures

Station 1: Segregate Equipment Drop

Deposit equipment used on-site (i.e., tools, sampling devices and containers, monitoring instruments radios, clipboards, etc.) on the plastic drop cloth/sheet or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross contamination. Loose leaf sampling data sheets or maps can be placed in plastic zip lock bags if contamination is evident.

Station 2: Physical Removal With A High-Pressure Washer (Optional)

Use high pressure wash on grossly contaminated equipment. Do not use high- pressure wash on sensitive or non-waterproof equipment.

Station 3: Physical Removal With Brushes And A Wash Basin

Scrub equipment with soap and water using bottle and bristle brushes. Only sensitive equipment (i.e., radios, air monitoring and sampling equipment) which is waterproof should be washed. Equipment which is not waterproof should have plastic bags removed and wiped down with a damp cloth. Acids and organic rinses may also ruin sensitive equipment. Consult the manufacturers for recommended decontamination solutions.

Station 4: Equipment Rinse

Wash soap off of equipment with water by immersing the equipment in the water while brushing. Repeat as many times as necessary.

Station 5: Low-Pressure Rinse

Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

Station 6: Nitric Acid Sprayers (required only if metals are a contaminant of concern)

Using a spray bottle rinse sampling equipment with nitric acid. Begin spraying (inside and outside) at one end of the equipment allowing the acid to drip to the other end into a 5-gallon bucket. A rinsate blank may be required at this station. Refer to Section 9.

Station 7: Low-Pressure Sprayers

Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

Station 8: Organic Solvent Sprayers

Rinse sampling equipment with a solvent. Begin spraying (inside and outside) at one end of the equipment allowing the solvent to drip to the other end into a 5-gallon bucket. Allow the solvent to evaporate from the equipment before going to the next station. A QC rinsate sample may be required at this station.

Station 9: Low-Pressure Sprayers

Rinse sampling equipment with distilled/deionized water with a low-pressure washer.

Station 10 : Clean Equipment Drop

Lay clean equipment on plastic sheeting. Once air dried, wrap sampling equipment with aluminum foil, plastic, or other protective material.

7.2.3 Post Decontamination Procedures

1. Collect high-pressure pad and heavy equipment decontamination area liquid and waste and store in appropriate drum or container. A sump pump can aid in the collection process. Refer to the Department of Transportation (DOT) requirements for appropriate containers based on the contaminant of concern.

2. Collect high-pressure pad and heavy equipment decontamination area solid waste and store in appropriate drum or container. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

3. Empty soap and water liquid wastes from basins and buckets and store in appropriate

drum or container. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

4. Empty acid rinse waste and place in appropriate container or neutralize with a base and place in appropriate drum. pH paper or an equivalent pH test is required for neutralization. Consult DOT requirements for appropriate drum for acid rinse waste.

5. Empty solvent rinse sprayer and solvent waste into an appropriate container. Consult DOT requirements for appropriate drum for solvent rinse waste.

6. Using low-pressure sprayers, rinse basins, and brushes. Place liquid generated from this process into the wash water rinse container.

7. Empty low-pressure sprayer water onto the ground.

8. Place all solid waste materials generated from the decontamination area (i.e., gloves and plastic sheeting, etc.) in an approved DOT drum. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

9. Write appropriate labels for waste and make arrangements for disposal. Consult DOT regulations for the appropriate label for each drum generated from the decontamination process.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITYASSURANCE/ QUALITY CONTROL

A rinsate blank is one specific type of quality control sample associated with the field decontamination process. This sample will provide information on the effectiveness of the decontamination process employed in the field.

Rinsate blanks are samples obtained by running analyte free water over decontaminated sampling equipment to test for residual contamination. The blank water is collected in sample containers for handling, shipment, and analysis. These samples are treated identical to samples collected that day. A rinsate blank is used to assess cross contamination brought about by improper decontamination procedures. Where dedicated sampling equipment is not utilized, collect one rinsate blank per day per type of sampling device samples to meet QA2 and QA3 objectives.

If sampling equipment requires the use of plastic tubing it should be disposed of as contaminated and replaced with clean tubing before additional sampling occurs.

10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results in accordance with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow OSHA, U.S. EPA, corporate, and other applicable health and safety procedures.

Decontamination can pose hazards under certain circumstances. Hazardous substances may be incompatible with decontamination materials. For example, the decontamination solution may react with contaminants to produce heat, explosion, or toxic products. Also, vapors from decontamination solutions may pose a direct health hazard to workers by inhalation, contact, fire, or explosion.

The decontamination solutions must be determined to be acceptable before use. Decontamination materials may degrade protective clothing or equipment; some solvents can permeate protective clothing. If decontamination materials do pose a health hazard, measures should be taken to protect personnel or substitutions should be made to eliminate the hazard. The choice of respiratory protection based on contaminants of concern from the site may not be appropriate for solvents used in the decontamination process.

Safety considerations should be addressed when using abrasive and non-abrasive decontamination

equipment. Maximum air pressure produced by abrasive equipment could cause physical injury. Displaced material requires control mechanisms.

Material generated from decontamination activities requires proper handling, storage, and disposal. Personal Protective Equipment may be required for these activities.

Material safety data sheets are required for all decontamination solvents or solutions as required by the Hazard Communication Standard (i.e., acetone, alcohol, and trisodiumphosphate).

In some jurisdictions, phosphate containing detergents (i.e., TSP) are banned.

12.0 REFERENCES

Field Sampling Procedures Manual, New Jersey Department of Environmental Protection, February, 1988.

A Compendium of Superfund Field Operations Methods, EPA 540/p-87/001.

Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, USEPA Region IV, April 1, 1986.

Guidelines for the Selection of Chemical Protective Clothing, Volume 1, Third Edition, American Conference of Governmental Industrial Hygienists, Inc., February, 1987.

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, October, 1985.

APPENDIX A

Table

Table 1. Soluble Contaminants and Recommended Solvent Rinse

TABLE 1 Soluble Contaminants and Recommended Solvent Rinse		
SOLVENT[1]	EXAMPLES OF SOLVENTS	SOLUBLE CONTAMINANTS
Water	Deionized water Tap water	Low-chain hydrocarbons Inorganic compounds Salts Some organic acids and other polar compounds
Dilute Acids	Nitric acid Acetic acid Boric acid	Basic (caustic) compounds (e.g., amines and hydrazines)
Dilute Bases	Sodium bicarbonate (e.g., soap detergent)	Acidic compounds Phenol Thiols Some nitro and sulfonic compounds
Organic Solvents [2]	Alcohols Ethers Ketones Aromatics Straight chain alkalines (e.g., hexane) Common petroleum products (e.g., fuel, oil, kerosene)	Nonpolar compounds (e.g., some organic compounds)
Organic Solvent[2]	Hexane	PCBs

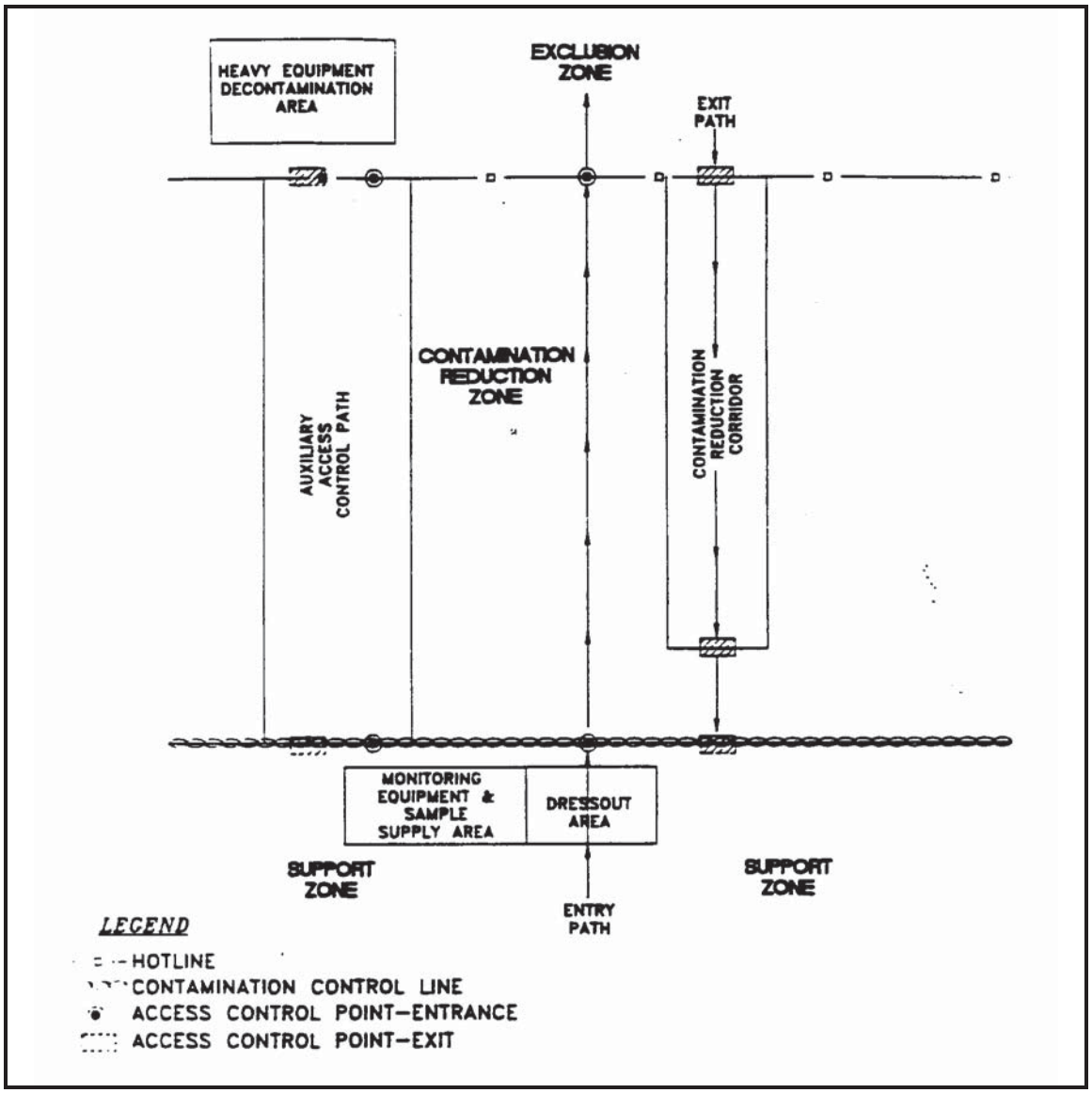
[1] - Material safety data sheets are required for all decontamination solvents or solutions as required by the Hazard Communication Standard

[2] - WARNING: Some organic solvents can permeate and/or degrade the protective clothing

APPENDIX B

Figures

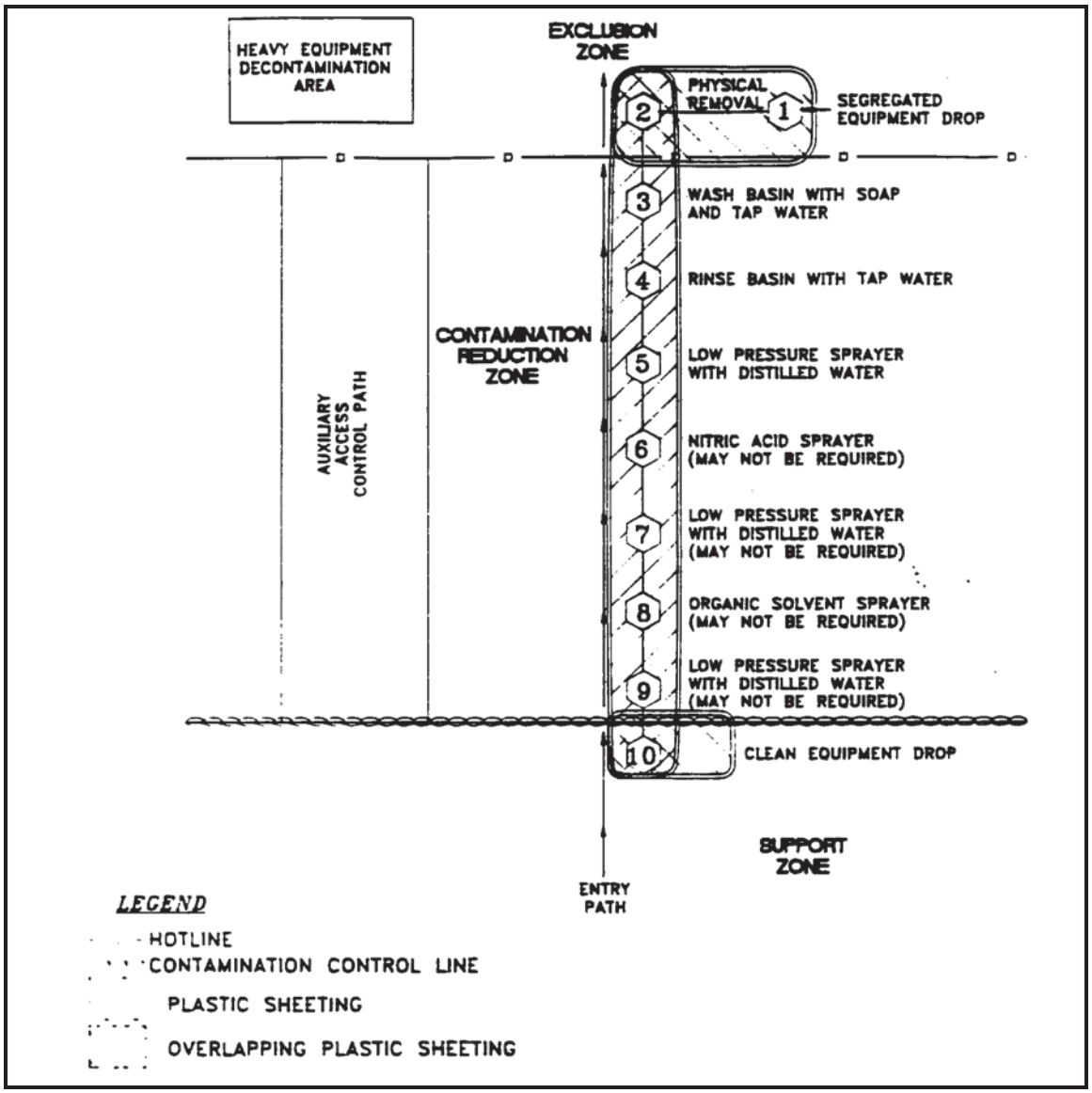
Figure 1. Contamination Reduction Zone Layout



APPENDIX B (Cont'd.)

Figures

Figure 2. Decontamination Layout



EPA SOP 2420.4C –
Field Chain of Custody for Environmental Samples,
December 2, 2003

STANDARD OPERATING PROCEDURE

No. 2420.4C

FIELD CHAIN OF CUSTODY FOR ENVIRONMENTAL SAMPLES

December 2, 2003

by Nicole Roblez

ENSV/RLAB/CATS

APPROVED:

Peer Reviewer — Date: 12/3/03

Manager, Regional Laboratory — Date: 4 Dec 03

Harold D. Brown — Date: 12/08/03

Independent QA Reviewer

Recertified

Reviewer					
Date					

TABLE OF CONTENTS

Attachments

A. Purpose

The purpose of this Standard Operating Procedure (SOP) is to establish uniform policies and procedures for use by field personnel to maintain an accurate written record of environmental samples from the time of collection through their acceptance by a laboratory for analysis. The custody procedures utilized within the laboratory for receiving samples and maintaining custody through the analytical processes are not covered in this SOP. See "Storage and Security of Environmental Samples", SOP 2420.2 for custody procedures utilized within the Regional Laboratory (RLAB).

B. Applicability

The policies and procedures outlined in this SOP are applicable to all Environmental Services Division (ENSV) personnel, Environmental Protection Agency (EPA), state/local agencies, and/or EPA contractors who collect environmental field samples for analyses by the RLAB or contract laboratories.

C. Summary of Procedures

As a requirement of any activity which may be used to support litigation proceedings, the validity of any data introduced into evidence must be clearly demonstrated. In the case of samples collected in support of an enforcement case, it must be clearly documented that the sample introduced into evidence is, in fact, the same sample collected and/or that the analytical data offered into evidence accurately represent the environmental conditions at the time of sample collection. It is imperative that there is adequate proof to demonstrate that transfer, storage or analysis, and that the analytical results were obtained from the same sample collected. Therefore, an accurate written record must be maintained to track the possession and handling Chain Of Custody Record (COC) (see Attachment 2) of each sample from the moment of collection through analysis and its introduction into evidence.

By definition, a sample is in "custody" if:

1. It is in one's actual physical possession; or
2. It is in one's view, after being in one's physical possession; or
3. It is locked up so no one can tamper with it, after being in one's physical possession; or
4. It is placed in a designated secured area

D. Definitions/Acronyms

ASR	Analytical Services Request
CLP	Contract Laboratory Program
COC	Chain of Custody Record

ENSV	Environmental Services Division
EPA	Environmental Protection Agency
LIMS	Laboratory Information Management System
PM	Project Manager
PO	Project Officer
QC	Quality Control
RECAP	Region 7 Environmental Collection and Analysis Program
ESAT	Environmental Services Assistance Team
RLAB	Regional Laboratory
RSCC	Regional Sample Control Coordinator
SOP	Standard Operating Procedure
SRN	Sample Receipt Notice
Tags	Sample container labels
UPS	United Parcel Service
VOA	Volatiles

E. Personnel Qualifications

Personnel performing this task should have a basic knowledge of the RLAB sample and records management procedures.

F. Responsibilities

1. Project Manager

a. The Project Manager submits a completed Analytical Services Request (ASR) to the RLAB 30 days before initiation of the sampling activity.

b. The Project Manager or designee (i.e., field contractor) ships and/or delivers properly collected, preserved, labeled, and packaged samples to the RLAB.

c. The Project Manager or designee (i.e., field contractor) is responsible for the accuracy and completeness of all accompanying paperwork. If any changes are required as a result of the sampling (e.g., sample number changes, additional analyses, samples not collected, quality control (QC) code additions), the Project Manager or designee (i.e., field contractor) must see that these corrections are made on all paperwork.

All changes made to the paperwork (COC, sample tags, or field sheets) must also be made to the information contained in the LIMS. It is the responsibility of the Project Manager or designee to supply correct information so that the Regional Sample Control Coordinator (RSCC) can

properly process the samples into the LIMS. Whenever possible, any changes are made prior to the delivery of the samples. If necessary, the RSCC will assist the Project Manager when changes are noted prior to sample collection/delivery, concurrent to sample delivery or after.

d. The Project Manager must be available to help resolve any problems with the samples or must designate someone to do this for them in their absence. This requires that when delivering samples, the Project Manager or designee stays with the RSCC to answer any questions. Samples must not be just dropped off (unless after normal business hours).

The Project Manager or designee calls the RSCC close to the anticipated delivery date and/or time that samples are sent by courier (i.e., Federal Express) to confirm that samples have arrived and to answer any questions the RSCC may have.

2. RSCC

a. The RSCC opens the ice chest (cooler) and utilizing the Infrared Digital Thermometer, checks the cooler temperature and records the temperature (in degrees Celsius) in the last row of the "Receiving Laboratory Remarks/Other Information" column on the COC (see Attachment 2).

b. The RSCC verifies the presence of all samples, checks all documentation and signs the COC after all paperwork is complete and accurate.

c. The RSCC works with the Project Manager to obtain correct information and puts the amended information into the LIMS.

d. The RSCC notifies the Project Manager of problems which prevent acceptance of the samples by ENSV. RLAB maintains all samples received in a secure location including those pending reconciliation of problems.

e. The RSCC logs samples into the LIMS and is responsible for the proper storage, tracking and/or distribution of the samples to the appropriate contract laboratories (this includes while the sample is in transit to the contract laboratory facility). The RSCC prepares an electronic Sample Receipt Notice (SRN) message for each activity received by the RLAB and routes it appropriately to the Environmental Services Assistance Team (ESAT), the Contract Laboratory Program (CLP) PO, or the Region 7 Environmental Collection and Analysis Program (RECAP) PO, CATS PM, ANOP PM, and appropriate back-up personnel.

G. Procedures

1. In order to ensure adequate control and documentation of collected samples, the number of personnel handling the samples from the time of collection through delivery to RLAB should be limited.

2. The following actions must be accomplished in order to ensure that the relationship between the physical sample and the description of the sample is clearly, completely and accurately established, and that the custody of the sample is initiated from the time of actual sample collection.

   a. A unique number is assigned to each sample (see "Identification, Documentation, and Tracking of Samples", SOP No. 2420.5) in order to relate the descriptive information to a physical sample. If a sample consists of several containers for analysis of different parameters from the same physical sample, the same number is used for each portion of the original sample.

   b. A sample tag (sample container label) is securely attached to each container at the time of collection for specific instructions for filling out the sample tag (see "Identification, Documentation and Tracking of Samples", SOP No. 2420.5).

   c. Custody of the sample is initiated at the time of collection by ensuring that the sample is in the sample collector's physical possession or view at all times, or is stored in a locked place where no one can tamper with it.

   The sample collector is responsible for the collected samples until they are delivered to the RLAB.

3. Samples may be delivered to RLAB by the sampler or EPA contractor via courier or commercial carrier.

   a. Sampler or EPA contractor-conveyed samples are those transported and delivered to RLAB. The coolers may be sealed or unsealed, but the sampler or EPA contractor must ensure that they are secured in the transport vehicle when he/she is not physically with the vehicle.

   b. Samples may be delivered via courier (e.g., Greyhound). The cooler and sample containers must be transported with the lids secured. The transfer of possession of the samples must be recorded from the sampler or EPA contractor to RLAB.

c. Samples may be shipped via commercial carrier (e.g., Federal Express, Airborne, United Parcel Service (UPS)) from the field to RLAB. The cooler and sample containers must be sealed at the time of shipment.

4. Samples are considered to be sealed when they are packaged in such a manner that would prohibit tampering or readily reveal any tampering, if it occurred.

a. A custody seal (see Attachment 1) may be used to secure the individual sample container, as appropriate to meet specific regulatory program requirements. These custody seals must be signed and dated by the sampler or EPA contractor when used to seal individual sample containers.

b. The use of a custody seal must be used to secure the openings of boxes, plastic bags, ice chests or coolers containing samples. These custody seals must be signed and dated by the sampler or EPA contractor when used to seal the shipping containers.

5. The COC (see Attachment 2) is initiated at the time of sample collection and must accompany all samples. The COC is utilized to document the transfer of a sample from the sampler or EPA contractor through receipt by the RSCC or designated back-up at RLAB.

RLAB instructions for the completion of the COC are outlined in Attachment 3.

a. The transfer of possession of the samples would occur when the sampler or EPA contractor delivers the samples to RLAB, gives them to the courier who will deliver the samples to RLAB, or packs the samples in a sealed shipping container for shipment to RLAB via commercial carrier.

b. The original and yellow copy of the COC will accompany the samples to RLAB. When the samples are conveyed by the sampler or EPA contractor, the COC may be hand carried. When the samples are delivered via courier or commercial carrier, the COC must be placed in a plastic document enclosure which is enclosed in the shipping container.

6. When samples are delivered to RLAB after duty hours, the samples and the COC will be placed in the refrigerator located on the back dock until acceptance by the RSCC or designated backup in accordance with the procedures outlined in "Storage and Security of Environmental Samples", SOP No. 2420.2.

7. Once RLAB has accepted the samples, the responsibility for custody of the samples transfers to the RLAB personnel. Custody of the samples is maintained through analysis in accordance with the laboratory's internal control procedures.

8. The original of the completed COC is obtained by RLAB for inclusion with the permanent site activity files, and included with the final data transmittal sent to the Project Manager.

9. The yellow copy of the completed COC is returned to the Project Manager for inclusion in their appropriate activity files after all samples, for a given activity, have been accepted.

10. The custody seals or evidence tape associated with the specific samples or sample shipments are not retained.

H. Quality Assurance/Quality Control

A written tracking record (COC) is maintained from the time that the sample is collected to its transfer from the collection site to its laboratory destination. This record is used to demonstrate that sample possession has been secured and limited. Signed and dated custody seals placed over the access points of the sample shipment demonstrate that the contents of the samples have not been tampered with or compromised.

I. References

1. US EPA, Region 7,"RLAB Procedures for Sample Receipt and Log-In", Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.1

2. US EPA, Region 7, "Identification, Documentation, and Tracking of Samples", Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.5

3. US EPA, Region 7, "Storage and Security of Environmental Samples", Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.2

Attachment 1

RLAB Custody Seal

Attachment 2

CHAIN OF CUSTODY RECORD
ENVIRONMENTAL PROTECTION AGENCY REGION VII

ACTIVITY LEADER(Print)	NAME OF SURVEY OR ACTIVITY	DATE OF COLLECTION DAY MONTH YEAR	SHEET of

CONTENTS OF SHIPMENT

SAMPLE NUMBER	TYPE OF CONTAINERS CUBITAINER	BOTTLE	BOTTLE	BOTTLE	VOA SET (2 VIALS EA)	SAMPLED MEDIA water	soil	sediment	dust	other	RECEIVING LABORATORY REMARKS/OTHER INFORMATION (condition of samples upon receipt, other sample numbers, etc.)
	NUMBERS OF CONTAINERS PER SAMPLE NUMBER										

DESCRIPTION OF SHIPMENT

_____ PIECE(S) CONSISTING OF _____ BOX(ES)

_____ ICE CHEST(S): OTHER _____

MODE OF SHIPMENT

_____ COMMERCIAL CARRIER: _____

_____ COURIER

_____ SAMPLER CONVEYED _____ (SHIPPING DOCUMENT NUMBER)

PERSONNEL CUSTODY RECORD

RELINQUISHED BY (SAMPLER)	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	

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Attachment 3

Instructions For Completing A Chain Of Custody Record

(Note: Each numbered item explains what is to be entered into that particular block moving from left to right, top to bottom of the document.)

1. Activity Leader. Enter the first initial and last name of the EPA Project Manager.

2. Name of Survey or Activity. Enter the activity number and/or Analytical Services Request (ASR) number (e.g., ERN07/900) for which the samples were collected.

3. Date of Collection. Enter the day, month, and year the samples were collected.

4. Sheet. Enter 1 of 1 unless there are more than one total sheets describing the shipment. If multiple sheets, enter the consecutive number of each sheet of the total number of sheets (e.g., 1 of 3, 2 of 3, 3 of 3).

5. Contents of the Shipment.

    a. Enter the specific sample numbers, number of sample type containers per sample number and sample media in the appropriate column

        (1) The ASR number and the individual sample numbers composing the shipment are entered in the "Sample Number" column (e.g., 2222-2). If more than one sheet is required, continue on additional sheets. For shipments of a large group of samples, it would be more appropriate and efficient to complete a separate sheet for each shipping container.

        (2) The types of containers for each sample number are entered in the columns provided. The size should be entered above the container type, as appropriate. For Volatiles, the "VOA Set" refers to two=40 ml vials contained in the cubitainer which are collected for volatile organics analyses. The container types are modified, as necessary or appropriate, to describe sample containers.

        (3) The sampled media for each sample number will be indicated by placing an "X" in the appropriate column. If the sample media is not listed, the actual media sampled should be entered in the "Other" column (e.g., wipe, sludge, air, biota, fish, etc.).

        (4) The "Receiving Laboratory Remarks/Other Information" is to be used by the RLAB to indicate any problems with the shipment or condition of the samples upon receipt; e.g., custody seal on sample container or shipping container broken, a sample container broken in transit, a sample lost due to leakage during shipment, etc. The temperature of the shipping coolers(s) are to be recorded in the lower area of this column. This column may also be used to record other sample numbers for cross-referencing purposes (e.g., external sample number).

b. After entering all of the above information, the total contents of the shipment should be indicated by marking out any remaining lines in this section. This can be accomplished either by drawing a line across the next line after the last entry and entering "None to Follow" or "Activity/ASR Complete," or by drawing a line across the next blank line or diagonally across the remaining lines in the section and entering "None to Follow" or "Activity/ASR Complete."

6. Description of Shipment. Enter the total number of pieces (e.g., samples or sample containers) packed in the total number of shipping containers (e.g., ice chests, boxes or other, which comprise the total shipment)(e.g., 12 pieces in 2 ice chests or 24 pieces in 2 boxes).

7. Mode of Shipment. Indicate the mode by which the samples are shipped to the RLAB by placing an "X" in the appropriate line preceding the specific mode in this block. If the shipment is via commercial carrier, the name of the carrier and the shipping document number (e.g., airbill) should be entered in the appropriate lines provided. This information may be entered by the sample shipper (sampler or individual to whom the sampler relinquished the samples), or the shipment receiver (lab sample custodian), as appropriate.

8. Personnel Custody Record. This portion of the form provides the record of changes of custody of the shipment (sample or group of samples)from the sample collector to the laboratory. To provide an adequate written record, all of the blocks should be completed as described below.

a. The sample collector will sign the first "Relinquished By" block when the samples are presented to another individual or commercial carrier.

(1) An "X" should be entered in the appropriate block to indicate whether the shipment is sealed or unsealed with a piece of completed custody seal tape, the date and time when the samples are relinquished should be entered in the appropriate blocks, and the reason for change of custody (e.g., transport to lab, receipt by lab, etc.) should be entered in the appropriate block.

(2) If the sampler is presenting the samples to a commercial carrier for shipment, the name of the carrier should be entered in the next available "Received By" block. The signature of a representative of the carrier is not required.

b. Each individual who received the shipment of samples will sign the next available "Received By" block and enter an "X" in the appropriate block to indicate whether the samples were received sealed or unsealed with a piece of completed custody seal tape. If the samples were shipped via commercial carrier, the individual receiving the samples (e.g., sample custodian at the RLAB) should enter the date and time the samples were received and the reason for change of custody (e.g., receipt by the RLAB) in the appropriate blocks.

c. Each successive individual who relinquishes custody of the samples will sign the next available "Relinquished By" block, enter an "X" in the appropriate block to indicate whether the sample shipment is sealed or unsealed with a piece of completed custody seal tape, enter the date and time when custody is relinquished and enter the reason for change of custody in the appropriate blocks.

Field Logbook Use and Maintenance	SOP No.: 4.07 SOP Category: HTRW Revision No.: 1 Date: December 2010

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the methods for use and maintenance of field logbooks. This procedure outlines methods, lists examples for proper data entry into a field logbook, and provides the standardized HydroGeoLogic, Inc. (HGL) format.

2.0 SCOPE AND APPLICATIONS

This procedure provides guidance for routine field operations on environmental projects. Site-specific deviations from the methods presented herein must be approved by the assigned HGL project manager and the HGL project quality assurance/quality control officer. Consult the project-specific planning documents for other documentation requirements that apply to the project.

3.0 GENERAL REQUIREMENTS

All work will be performed in a manner that is consistent with Occupational Safety and Health Administration established standards and requirements. Refer to the site- or project-specific health and safety plan for relevant health and safety requirements.

Personnel who use this procedure must provide documented evidence to the program manager or project manager that they have been trained on the procedure. This documentation will be retained in the project file.

Any deviations from specified requirements will be justified to and authorized by the project manager and/or the relevant program manager and documented in the planning documents. Deviations from requirements will be sufficiently documented to re-create the modified process.

All field personnel who travel to a site to conduct work related to environmental projects are responsible for documenting field investigation activities in project field logbooks in a legible manner and maintaining field logbooks over the course of the project in accordance with this SOP. Daily logs will be kept during field activities by the HGL field team leader, or approved designee, to provide daily records of significant events, observations, and measurements taken in the field.

The project manager or an approved designee is responsible for checking the field logbooks and verifying that they have been completed in accordance with this SOP.

4.0 PROCEDURE

4.1 INTRODUCTION

Field logbooks provide a means for recording observations and activities at a site. Field logbooks are intended to provide sufficient data and observation notes to enable participants to reconstruct events that occurred while performing field activities and to refresh the memory of field personnel when writing reports or giving testimony during legal proceedings. As such, all entries will be as factual, detailed, and as descriptive as possible so that a particular situation can be reconstructed without reliance on the collector's memory. Field logbooks are not intended to be used as the sole source of project or sampling information. A sufficient number of logbooks will be assigned to a project to ensure that each field team has a logbook at all times.

4.2 FIELD LOGBOOK IDENTIFICATION

Field logbooks shall be bound books with consecutively numbered pages. Logbooks will be permanently assigned to field personnel for the duration of a project, or sampling event. When not in use, the field logbooks are to be stored in site project files. If site activities stop for an extended period of time (2 weeks or more), field logbooks will be stored in the project files in the appropriate HGL office.

The cover of each logbook will contain the following information:

- Organization to which the book is assigned (HGL)
- Project number (if different than site number)
- Book number
- Site name

4.3 LOGBOOK ENTRY PROCEDURES

Every field team will have a logbook, and each field activity will be recorded in the logbook by a designated field team member to provide daily records of significant events, observations, and measurements during field operations. Beginning on the first blank page and extending through as many pages as necessary, the following list provides examples of useful and pertinent information that may be recorded (optional).

- Serial numbers and model numbers for equipment that will be used for the project duration
- Formulas, constants, and example calculations
- Useful telephone numbers

- County, state, and site address

Entries into the logbook may contain a variety of information. At a minimum, logbook entries must include the following information at the beginning of each day:

- Date
- Site name, site location, and project number
- Start time
- Weather
- All field personnel and subcontractors present and directly involved
- Level of personal protective equipment being used on the site
- Equipment used and calibration procedures followed
- Any field calculations

In addition, information recorded in the field logbook during the day will include, but is not limited to, the following:

- Sample description including sample numbers, time, depth, volume, containers, preservative, and media sampled
- Information on field quality control samples (e.g., duplicates)
- Sample courier airbill numbers and associated chains-of-custody
- Observations about site and samples (odors, appearances, etc.)
- Information about any activities, extraneous to sampling activities, that may affect the integrity of the samples
- Any public involvement, visitors, or press interest, comments, or questions; as well as times present at site
- Equipment used on site including time and date of calibration along with calibration gas/fluid lot numbers and expiration dates
- Background levels of each instrument and possible background interferences
- Instrument readings for the borehole, cuttings, or samples in the breathing zone and from the specified depth of the borehole, etc.
- Field parameters (pH, specific conductivity, etc.)
- Unusual observances, irregularities, or problems noted on site or with instrumentation used

- Maps or photographs acquired or taken at the sampling site, including photograph numbers and descriptions
- A description of the investigation-derived waste (IDW) generated, the quantity generated, and the manner of IDW storage employed
- A photograph log that lists subject and persons, distance to subject, person taking photograph, distance, direction, time, photograph number, and noteworthy items for each photograph
- Forms numbers and any information contained therein used during sampling (Note that a form does not take the place of the field logbook.)

All logbook entries will be made in indelible black or blue ink. No erasures are permitted. If an incorrect entry is made, the data will be crossed out with a single strike mark and initialed and dated by the originator. Entries will be organized into easily understandable tables if possible. A sample format is shown in Attachment 1.

All logbook pages will be initialed and dated at the top of each page. Times will be recorded next to each entry. No pages or spaces will be left blank. If the last entry for a day is not at the end of a page, a diagonal line will be drawn through the remaining space and the line will be initialed and dated.

Logbooks can become contaminated when used in the field. Every effort should be made by the field team to avoid contaminating the logbook. Logbooks can be kept in seal-top poly bags or temporary plastic covers may be used.

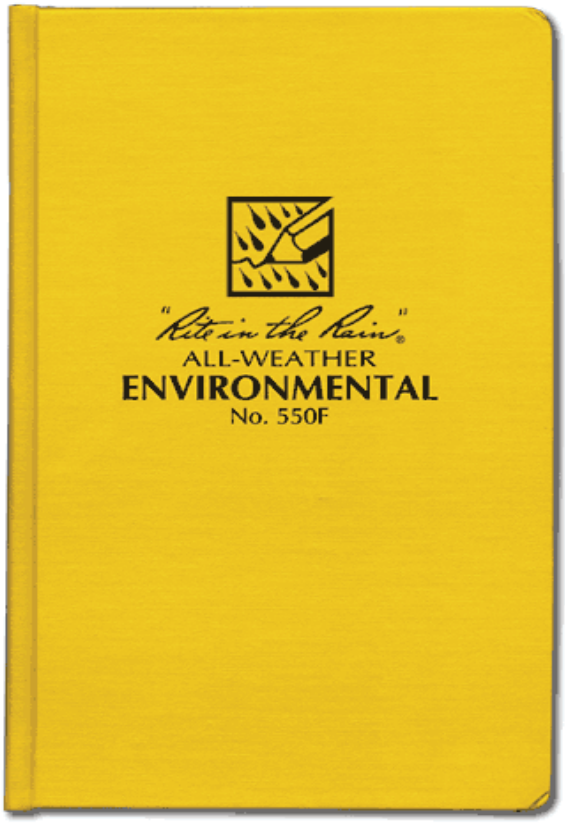
4.4 REVIEW

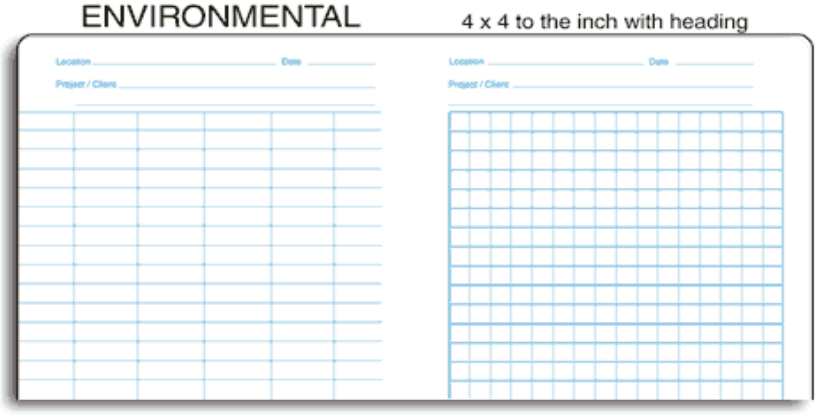
The assigned project leader or an approved designee will check field logbooks for completeness and accuracy on an appropriate site specific schedule determined by the project leader. Any discrepancies in these documents will be noted and returned to the originator for correction. The reviewer will acknowledge that these review comments have been incorporated by signing and dating the applicable reviewed documents.

5.0 ATTACHMENTS

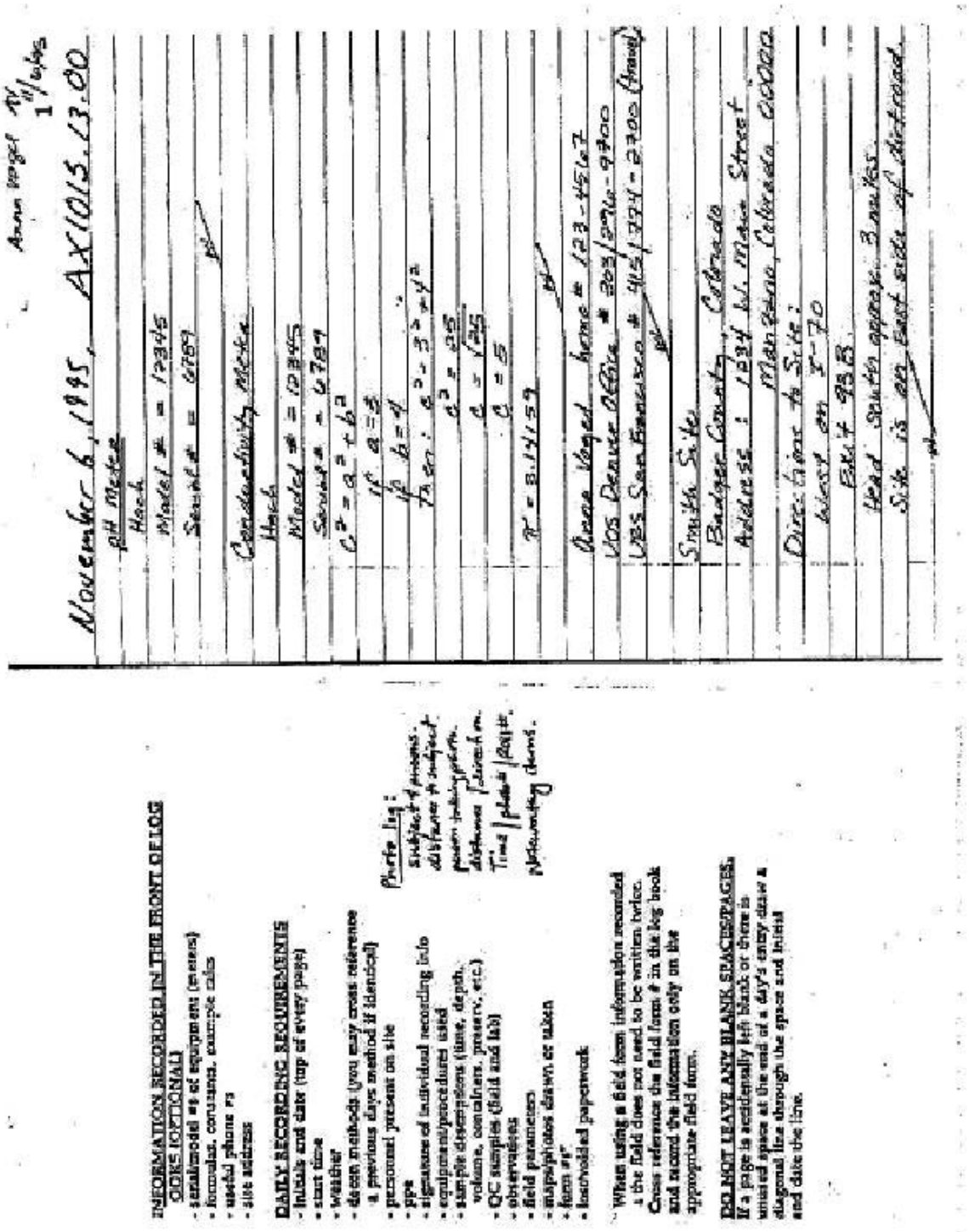
Attachment 1 Example Field Logbook

ATTACHMENT 1
Example Field Logbook

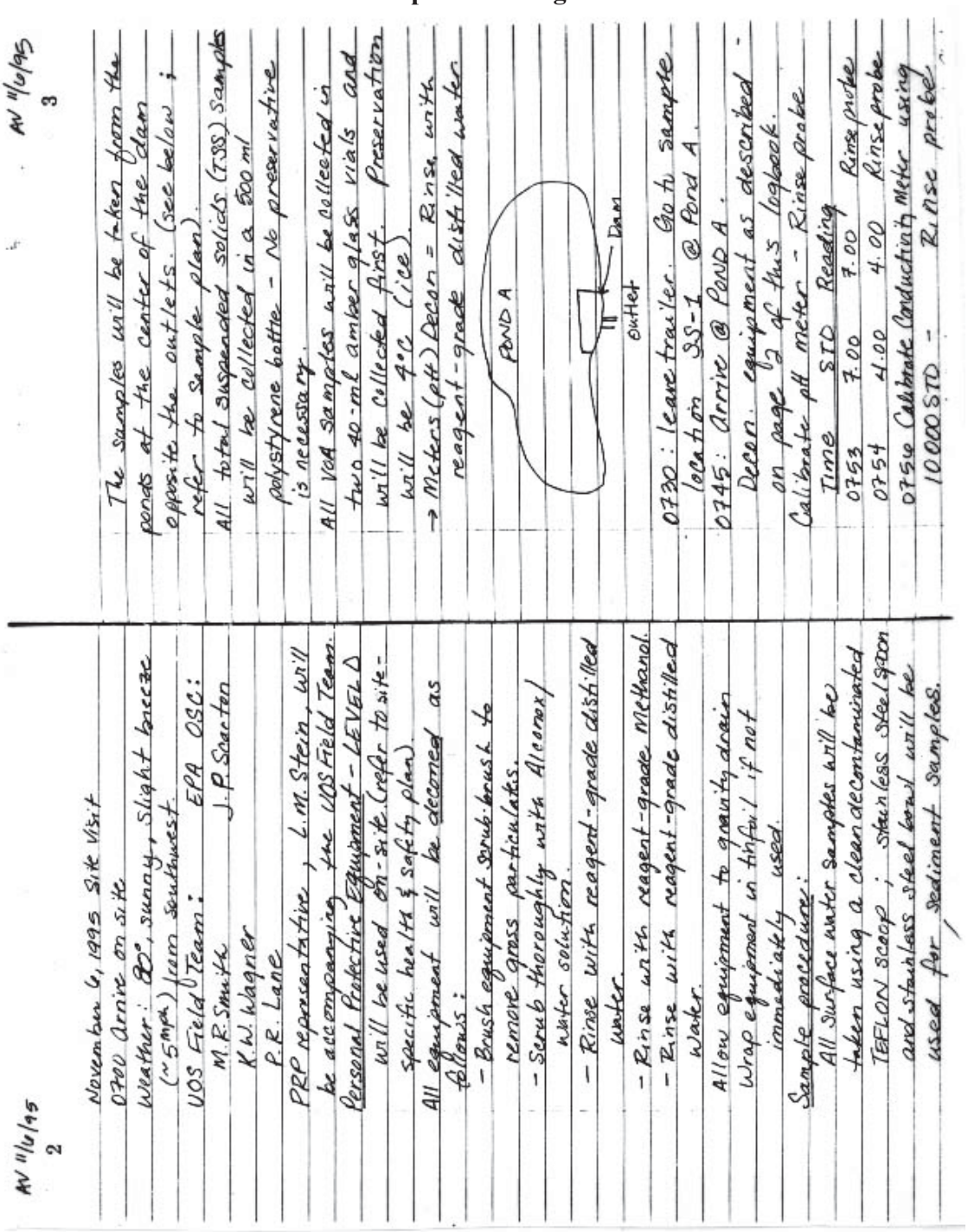




ATTACHMENT 1 (continued)
Example Field Logbook



ATTACHMENT 1 (continued)
Example Field Logbook



AV 11/6/95 2

November 6, 1995 Site Visit
0700 Arrive on site
Weather: 80°, sunny, slight breeze (~5mph) from southwest.
UOS Field Team: EPA OSC:
M.R. Smith J.P. Starten
K.W. Wagner
P.R. Lane
PRP representative, L.M. Stein, will be accompanying the UOS Field Team.
Personal Protective Equipment - LEVEL D
will be used on-site. (refer to site-specific health & safety plan)
All equipment will be decomed as follows:
- Brush equipment scrub brush to remove gross particulates.
- Scrub thoroughly with Alconox/ water solution.
- Rinse with reagent-grade distilled water.
- Rinse with reagent-grade Methanol.
- Rinse with reagent-grade distilled water.

Allow equipment to gravity drain
Wrap equipment in tinfoil if not immediately used.
Sample procedure:
All surface water samples will be taken using a clean decontaminated TEFLON scoop; stainless steel spoon and stainless steel bowl will be used for sediment samples.

The samples will be taken from the ponds at the center of the dam opposite the outlets. (see below; refer to Sample plan).
All total suspended solids (TSS) samples will be collected in a 500 ml polystyrene bottle - No preservative is necessary.
All VOA samples will be collected in two 40-ml amber glass vials and will be collected first. Preservation will be 4°C (ice).
→ Meters (pH) Decon = Rinse with reagent-grade distilled water

POND A
Dam
OUTLET

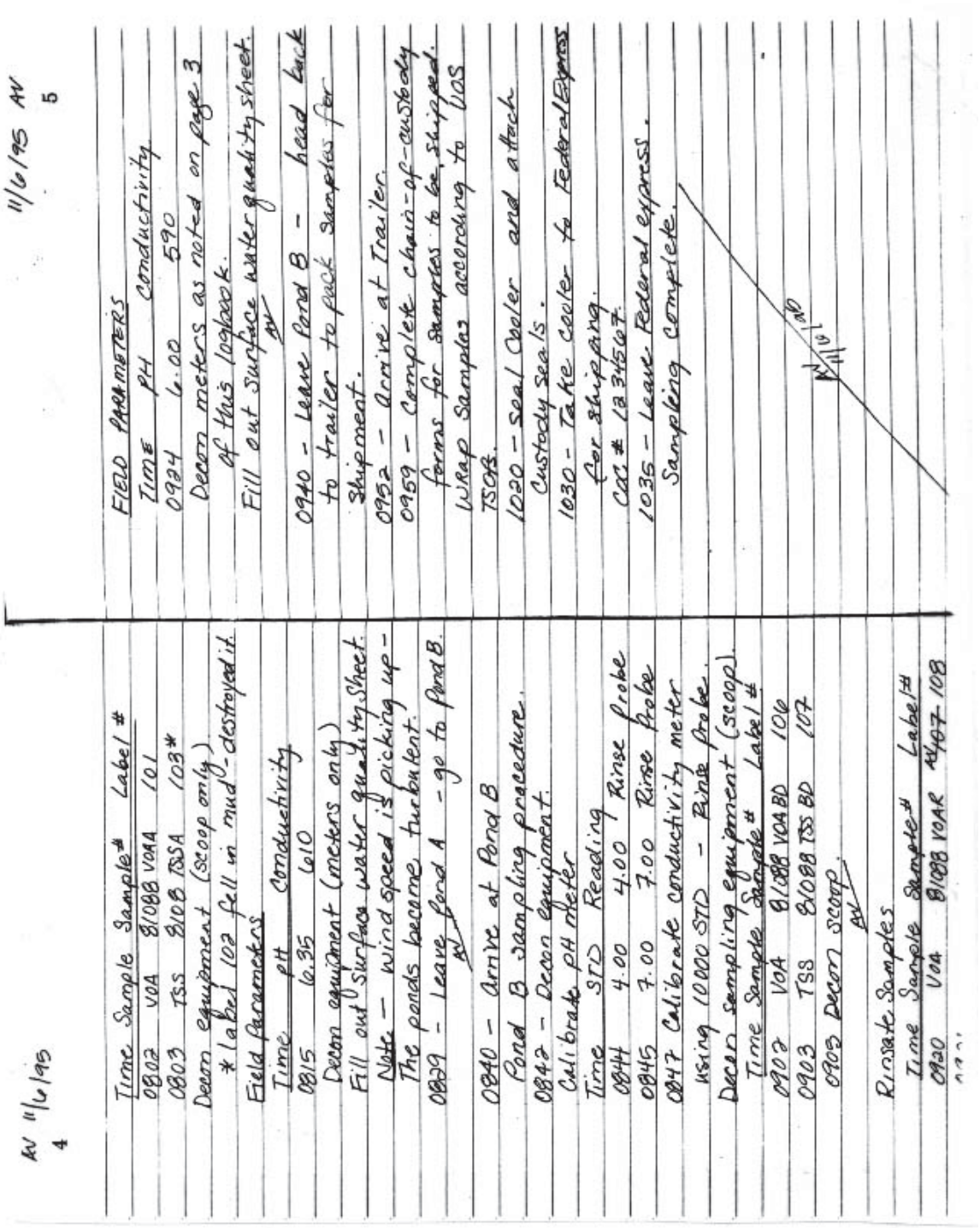
0730: Leave trailer. Go to sample location SS-1 @ Pond A.
0745: Arrive @ Pond A.
Decon. equipment as described - on page 2 of this logbook.
Calibrate pH meter - Rinse probe

Time	STD	Reading	
0753	7.00	7.00	Rinse probe
0754	4.00	4.00	Rinse probe

0756 Calibrate Conductivity Meter using 10,000 STD - Rinse probe

AV 11/6/95 3

ATTACHMENT 1 (continued)
Example Field Logbook



11/6/95 AV 5

FIELD PARAMETERS

Time	PH	Conductivity
0924	6.00	590

Decon meters as noted on page 3 of this logbook.

Fill out surface water quality sheet. AV

0940 - Leave Pond B - head back to trailer to pack Samples for Shipment.

0952 - Arrive at Trailer.

0959 - Complete Chain-of-custody forms for Samples to be shipped. Wrap Samples according to USS TSOP.

1020 - Seal Cooler and attach Custody Seals.

1030 - Take Cooler to Federal Express for shipping.

CAC # CA 346567.

1035 - Leave Federal express. Sampling complete.

AV 11/6/95 4

Time	Sample Sample#	Label #
0802	VOA	B1088 VOAA 101
0803	TSS	B108 TSSA 103*

Decon equipment (scoop only)

* Label 103 fell in mud - destroyed it.

Field Parameters

Time	pH	Conductivity
0815	6.35	6610

Decon equipment (meters only)

Fill out Surface water quality Sheet.

Note - wind speed is picking up - The ponds became turbulent.

0839 - Leave Pond A - go to Pond B. AV

0840 - Arrive at Pond B

Pond B sampling procedure.

0842 - Decon equipment.

Calibrate pH meter

Time	STD	Reading
0844	4.00	4.00 Rinse probe
0845	7.00	7.00 Rinse probe

0847 Calibrate conductivity meter using 10,000 STD - Rinse probe.

Decon sampling equipment (scoop).

Time	Sample Sample#	Label #
0902	VOA	B1088 VOA BD 106
0903	TSS	B1088 TSS BD 107

0905 Decon scoop. AV

Rinsate Samples

Time	Sample Sample#	Label/#
0920	VOA	B1088 VOAR AV07-108

STANDARD OPERATING PROCEDURE

4220.03A

Protocols for the Region 7 Lead-Contaminated Residential Yard Soil Cleanup Actions Procedures and Sequencing

June 8, 2007

Mark Doolan
SUPR/FFSE

APPROVED:

OSC Peer Review	Date
Risk Assessor Peer Review	Date
Manager, Federal Facilities/Special Emphasis Branch	Date
Manager, Enforcement/Fund-Lead Removal Branch	Date
Manager, Emergency Response & Removal Branch	Date
Independent QA Reviewer	Date

Recertified:

Branch/Name					
Date					

Table of Contents

A. PURPOSE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the procedures and the sequencing for residential yard soil cleanup at lead-contaminated sites. Prior to any yard soil cleanup actions, the sampling activities described in Section E.1 shall be conducted to enable appropriate decisions and sequencing for subsequent cleanup activities described in Section E.3.

B. SUMMARY OF METHOD

The major category of sites where sampling will be performed includes, but is not limited to active/former lead mining, milling and smelter sites, areas impacted by mining, milling, and smelter activities, mining depositories, transportation routes from mining, milling and smelter sites and the use of mining wastes in public and residential areas.

1. Soil

Soil samples may be collected using a variety of methods and equipment depending on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Surface soils may be easily sampled using a spade, trowel, and scoop.

2. Interior Dust

The amount of lead in interior dust samples can be expressed as a loading or as a concentration. Loading is the weight of lead per area sampled and the typical units are micrograms per sq cm ($\mu g/cm^2$). Concentration is the weight of lead per weight of sample and is typically reported as µg/g. Vacuum dust collection is able to generate both loading and concentration results. Both loading and concentration is required for residential site investigations.

In each residence, it is anticipated that three interior dust samples will be collected. Since each residence will have a different floor plan and furniture arrangement, it will not be possible to predetermine the exact sample location. Interior dust samples will be collected by wipe sampling and/or by the use of a high efficiency particulate air (HEPA) vacuum.

Wipe samples are collected from smooth surfaces to indicate surficial contamination; a sample location is measured and marked off. While wearing a new pair of surgical gloves, a sterile gauze pad is opened, and soaked with solvent. The solvent used is dependent of the surface being sampled. This pad is then stroked firmly over the sample surface, first vertically, then horizontally, to ensure complete coverage. The pad is then transferred to the sample container. All wipe sampled will be submitted for laboratory analysis of arsenic, cadmium, lead, and barium by EPA Method 6020. Contaminant levels will be expressed in $\mu g/cm^2$, which will represent the contaminant loading at each sampling location.

Interior dust samples from floors shall be collected accordance with Paragraph 11.11 and 11.2 of ASTM D543805 contained in Attachment A. Samples will be submitted for laboratory analysis of arsenic, cadmium, lead, and barium by EPA Method 6020. The contaminant levels will be expressed in µg/kg, which will represent the total concentration of each contaminant, and µg/cm^2, which will represent the contaminant loading at each sampling location.

3. Lead-Based Paint

Lead-Based paint (LBP) screening shall be conducted on the interior and exterior of homes at properties where dust samples are collected. The purpose of the LBP screening is to determine whether LBP is present in the home at a level that may create an exposure risk. It is not intended to be a comprehensive as a LPB inspection or a lead hazard screen as defined in the EPA regulations at 40 CFR 745.227.

Lead-Based paint (LBP) screening shall be conducted on the interior and exterior of homes at properties where dust samples are collected at Superfund lead-contaminated sites.

For exterior LBP, the paint chips will be collected with the soil sample and analyzed with an X-Ray Fluorescence (XRF) spectrometer. For interior LBP, an XRF spectrometer will be used to collect the reading from each painted surface that is selected.

C. DEFINITIONS

Residential properties: As defined in the Superfund Lead-Contaminated Residential Sites Handbook (Handbook) are any areas with high accessibility to sensitive populations, and includes properties containing single- and multi-family dwellings, apartment complexes, vacant lots in residential areas, schools, day-care centers, community centers, playgrounds, parks, green ways, and any other areas where children may be exposed to site-related contaminated media.

X-Ray Fluorescence (XRF) spectrometer: An instrument used to resolve radiation into spectra to determine measurements. Will be used to analyze soils for metals contamination as described in the Instruction Manual for the XRF spectrometer.

Integrated Exposure Uptake Biokinetic Model (IEUBK): Predicts blood-lead concentrations (PbBs) for an individual child, or group of similarly exposed child (6 months to 7 years old), who are exposed to lead in the environment.

D. PERSONNEL QUALIFICATIONS

All field personnel are required to take the 40-hour health and safety training course (as per 29 CFR 1910.120(b)(4)) and regular refresher courses prior to engaging in any field collection activities.

E. PROCEDURAL STEPS

1. Initial Site Investigation and Sampling Requirements

Prior to any yard soil cleanup actions, the sampling activities described in this section should be conducted to enable appropriate decisions and sequencing for subsequent cleanup action. The information collected during the initial sampling phase should be sufficient to enable the risk assessor to conduct a risk assessment and determine a risk-based cleanup level using the Integrated Exposure Uptake Biokinetic (IEUBK) Model. Additionally, sufficient sampling should be conducted in each individual residential property, such that no other additional sampling would be required prior to cleanup actions. All sampling should be conducted in accordance with the "Superfund Lead-Contaminated Residential Sites Handbook, OWSER 9258.7-50, May 2003" and as further defined in this directive.

Yard Soil: Initial sampling should be conducted to identify the total extent of contamination at the site. During the initial phase of the site investigation, the site boundary should be defined at the lead contamination concentration of 400 parts per million (ppm). If economically feasible, each and every individual residential property within the site should be sampled, beginning at the source and progressing outward until a clear outer boundary of the contamination is defined, and each residence within the boundary has been sampled. At large sites where funding does not allow the sampling of every property within the 400 ppm boundary during the initial investigations, the extent of lead contamination exceeding 1,200 ppm must, at a minimum, be defined. Every property within the 1,200 ppm boundary should be sampled along with a sufficient number of properties to determine the general location of the 400 ppm boundary.

Fine Soil Fraction Analysis: The IEUBK guidance requires that soil lead concentrations input to the model be only for the soil fraction smaller the 250 µm. In order to generate this required information, approximately 30 soil samples collected from site must be sieved through a 250 µm screen prior to analysis. Additionally, for risk assessment purposes, a correlation between the fine fraction and total soil must be established. The set of 30 sampled to be sieved must be analyzed as a whole sample first, then sieved through the 250 µm screen. The minus 250 µm size fraction will then be analyzed for comparison to the whole sample. A correlation between the fine fraction and total sample must then be developed for the site. If the 30 samples initially collected do not provide a statically significant correlation, additional samples must be collected until a correlation is determined. This correlation must then be used when setting the cleanup value based on total sample analysis.

Interior Lead-Based Paint/Interior Dust: The lead content of interior lead-based paint, and the lead loading and concentration of interior dust should be determined at a statistically significant number of homes, generally 20 percent. This assessment is required for both use in the risk assessment and for determining cleanup requirements.

Exterior Lead-Based Paint: Lead concentrations in exterior paint should be analyzed, and the general condition of exterior surfaces should be assessed to determine the potential for recontamination of the clean soil placed in the yards during the cleanup. Measurement of the lead concentration in exterior paint should be made at each home were yard soil samples are obtained.

High Child Impact Areas: Daycares, school properties and playgrounds, parks, and green spaces are all areas where large numbers of young children concentrate. All of these types of facilities and areas shall be sampled during the initial investigation at a site since these are the locations that may present exposure risk to large numbers of children. Sampling in these areas should be conducted, as described below for residential properties.

Bioavailability Testing: The IEUBK Model requires the input of the bioavailability of site soil. Samples of soil should be collected and analyzed for bioavailability using the in-vitro method from a statistically significant number of yards for risk assessment purposes. Generally, only 30 samples are required from the site for this purpose and should be collected from soil with varying lead concentrations from low to high.

TCLP: Soil samples should collect and analyze soil for TCLP from a statistically significant number of yards to determine disposal and/or pre-disposal treatment requirements. Sufficient samples must be collected at varying concentration levels to confidently determine the level where the soils will likely fail TCPL for lead.

Speciation Testing: If warranted by enforcement action against a potentially responsible party, or if there is doubt concerning the lead source, a soil-lead speciation study should be conducted by micro-probe analytical methods. Generally 20 to 30 samples collected throughout the site will fulfill this need.

Residential Water Wells: Sample all private residential water wells within the suspected boundaries of the site to determine the contribution of lead and other metals to overall risk.

2. Sampling Procedures

2.1 Soil Sampling Procedures

All soil sampling procedures should follow the guidance presented in the Superfund Lead-Contaminated Residential Sites Handbook (Handbook). Residential yards should be divided into quadrants based on the size of the yard. Composite samples comprised of five aliquots should be collect from each quadrant. The guidance from the Handbook is summarized below.

Yard Soil Samples: Residential lots with a total surface area less than 5,000 square feet will be divided into two quadrants; front yard and back yard. Aliquots comprising the front and back yard composites shall be equally spaced within the respective portion of

the yard, and should be collected outside of the drip zone and away from influences of any other painted surfaces. Composites shall consist of aliquots collected from the same depth interval.

For residential lots with a total surface area greater than 5,000 square feet, the property should be divided into four quadrants of roughly equal surface area. The two quadrants in the front yard should encompass one half of the side yard; likewise for the two quadrants in the back yard. One five-aliquot composite shall be obtained from each quadrant.

Any properties over one acre in size, including very large residential yards, vacant lots, schools, or parks, should be divided into 1/4 acre sections. One five aliquot composite sample shall be collected from each section. The aliquots shall be equal spaced and collected away from influences of the drip zone and any other painted surfaces. Composite samples must be comprised of aliquots from the same depth interval.

Drip Zones: A four-aliquot composite sample shall be collected from the drip zone of each residential property, regardless of lot size. The composite sample shall consist of a minimum of four aliquots collected between 6 and 30 inches from the exterior walls of the house. Each aliquot should generally be collected from the midpoint of each side of the house. Collection of additional aliquots should be considered if other factors exist, such as bare spots, distinct differences in the house exterior, and areas where runoff collects such as in downspout discharge areas.

Play Areas, Gardens, and Driveways: Distinct play areas and vegetable gardens, if present, should be sampled separately as discrete areas of the yard. Samples should also be collected in other locations depending upon the potential for exposure , such as under porches or crawl spaces and gravel driveways.

Depth Sampling: In order to determine the depth of lead contamination throughout the site, and to select the appropriate cleanup depths, samples shall be collected at depth from a representative number of homes throughout the site. Generally, 20 to 30 homes, equally spaced across the site, should be sampled to a depth of 24 inches. Individual composites shall be collected from each discrete quadrant at a depth of 0-1 inch; 0-6 inches; 6-12 inches; 12-18 inches; and 18-24 inches. The 0 to 1 inch sample, which is used to determine whether a property qualifies for cleanup, should focus on shallow surface soil which represents exposure.

Confirmatory Samples: Most field sampling for metals in residential properties is conducted using field portable X-Ray fluorescence (XRF) instruments. Laboratory confirmation samples shall be collected at a rate of five percent during the sampling event and submitted to the laboratory for wet chemistry analysis to confirm the results of the XRF. The XRF determined values shall be considered valid if the laboratory analytical results of the same sample are within plus or minus 30 percent of the XRF results. For decision making purposes where both an XRF and laboratory result was obtained for a sample, the higher value of the two will be used. For example, if the XRF recorded a

result of 390 ppm lead for a sample from a yard quadrant and the laboratory recorded 425 ppm for the same sample, the XRF sample result will be considered valid but the quadrant will be assigned a lead value of 425 ppm.

Sample Collection: Once the yard has been divided into quadrants, a five-aliquot composite sample shall be collected from each quadrant. The aliquot shall be collected from the same depth interval with a disposable or stainless steel spoon or trowel. The soil from each aliquot shall be placed into a pan and thoroughly mixed. Then three XRF measurements shall be obtained from the sample. The three readings shall be averaged to obtain the metals concentration for that quadrant. All three reading should be within 10 percent of each other. If any of the three reading falls outside the 10 percent range, the sample must be re-mixed and the procedure repeated until the criteria is achieved. If a laboratory confirmation sample is to be obtained from the quadrant, a portion of the sample should be placed into the XRF specimen cup after the three readings have been obtained and averaged. The XRF will then be used to analyze the specimen cup, and the reading will be recorded. The specimen cup shall then be submitted to laboratory for wet chemistry analysis. Comparison of the XRF and laboratory analysis shall be made between the lab data and the XRF reading of the specimen cup.

2.2 Dust Sampling Procedures

The amount of lead in dust samples can be expressed as a loading or as a concentration. Loading is the weight of lead per area sampled and the typical units are $\mu g/ft^2$. Concentration is the weight of lead per weight of sample and is typically reported as µg/g. Vacuum dust collection is able to generate both loading and concentration results. Both loading and concentration is required for residential site investigations. Three dust samples shall be collected from each residence scheduled for assessment. The general sample area with a description of sample location criteria is presented below. Dust samples from floors shall be collected in accordance with Paragraph 11.1 and 11.2 of ASTM D5438-05.

Entry Way: A vacuum sample will be collected from the most frequently used entry way to the residence. The sample location must be at least 3 feet away from the door. If there is an option between a hard floor surface and a carpeted floor surface, the hard floor surface area shall be chosen over the carpeted surface due to the potential for better sample collection. The sample will then be collected using the appropriate vacuum method for the floor type.

Floor: A sample of floor dust shall be collected from the most commonly used room in the residence other than a bedroom. The room, other than the bedroom, where children living in the home spend the most time on the floor in the room shall be chosen for the sample collection. If no children live at the residence, the room where residents spend the most time will be chosen. Sample location shall be based on the floor type in the room. Hard floor surface should be given preference over carpeted areas in the room. A sample location that is not in the main walking pathway of the room, and is also large enough to accommodate the sampling requirements, will be chosen as the sample location.

Bedroom: A sample shall be collected from one bedroom in the residence. If there are children living at the residence, the youngest child's bedroom shall be selected for sampling. If there are no children living at the residence, the bedroom where the most time is spent shall be selected. If a child's room is selected, regardless of floor type, the sample location shall be chosen based on where the child's play area is in the room or where they spend the most time on the floor in the room. If an adult bedroom is selected, the sample shall be collected based on floor type and a hard floor surface should be given preference over a carpeted floor.

2.3 Lead-Based Paint Assessment

Lead-based paint (LBP) screening shall be conducted on the interior and exterior of homes at properties where dust samples are collected. The purpose of the LBP screening is to determine whether LBP is present in the home at a level that may create an exposure risk. It is not intended to be as comprehensive as a LBP inspection or a lead hazard screen as defined in the EPA regulations at 40 CFR 745.227.

LBP readings shall be taken using an XRF instrument capable of provide data in milligrams per square centimeter (mg/cm^2) and capable of analyzing lead to less than 1 mg/cm^2. The following procedures will be followed during the LBP screening assessments.

An initial visual inspection shall be conducted of the exterior walls of the home and the interior painted surfaces in rooms where dust samples are collected to assess whether significant deteriorating paint is present. If significant deteriorating painted surfaces are observed on the exterior walls of the residence, each of the exterior walls of the residence will be analyzed for LBP. If significant deteriorating painted surfaces are observed in the interior rooms where the dust samples are collected, each of the four walls in the room and a minimum of two window sills shall be analyzed. The XRF readings shall be taken at the location of the deteriorating painted surfaces. If deteriorating painted surfaces are not observed on the exterior walls of the residence, each of the four walls of the residence will be analyzed for LBP using a XRF instrument. If deteriorating paint is not observed in the rooms where dust samples are collected, XRF readings will be taken from each of the four walls and a minimum of two window sills. The sampling team will document the general description of the interior walls and window sills in the rooms where XRF readings are taken.

3. Residential Yard Soil Cleanup

3.1 Initial Actions/Time-Critical Removal

The following actions should be initiated as soon practical after the information listed above is gathered and an unacceptable risk has been determined either through a risk assessment or the identification of a significant number of properties that exceed EPA's

time-critical removal action level of 1,200 ppm lead. Several of these actions do not involve soils cleanup, but are necessary to rapidly begin to control exposure.

Blood-lead monitoring: The project manager should immediately coordinate with the local or county health department(s) to initiate or enhance blood-lead monitoring of young children with the goal of 100% participation. Funding should be provided, if needed by the department, to begin or enhance the program. Agreements between the health departments and the Agency must be executed to allow the release of information on children identified with elevated blood-lead concentration for inclusion in cleanup actions.

Health Education: Like blood-lead monitoring, coordination with local/county health department and existing citizens groups to initiate health education programs in the community should begin immediately. Funding will likely need to be provided to these entities to conduct the program, and can usually be combined with funding provided for blood-lead monitoring.

HEPA Vacuums: If the sampling results indicate there is a significant exposure risk for interior dust, a vacuum loan program should be Initiated at the site. This program is also usually combined with, and is part of, the health education program funded through the local health department. Establishing this program early in project can aid in significantly reducing the exposure risk at the site until all source soils have be completely addressed.

Soil Disposal: The project manager must identify soil disposal options and establish the yard soil disposal facility prior to any excavation activities. Off-site soil disposal other than in a permitted RCRA Subtitle C and/or D land fill will require that a RCRA Remedial Action Plan is prepared and issued RCRA. Consideration should be given to innovative on-site disposal methods, such as deep fill for construction projects, capping of mining wastes, or other uses where exposure of young children is mitigated.

Yard Soil Removal: If the yard sampling results indicate a manageable number of contaminated homes exist on the site and can all be cleaned up within the removal budget, the cleanup of all residential yards where lead exceeds 400 ppm (or the site specific action level) should be completed. At large sites where the excavation of all residential yards over 400 ppm lead is not possible within the removal budget, the time-critical removal action should address all residences where soil-lead exceeds 1,200 ppm, and all residences where soil-lead exceeds 400 ppm and a child identified with high blood-lead resides.

High Child Impact Areas: All soil exceeding 400 ppm lead, or the site specific action level, shall be excavated for the HCIAs within the site. Priority should be given to the HCIAs, especially day care facilities, for cleanup early in the remediation project since these areas can affect the largest number of young children at the site.

Exterior Lead-Based Paint: For residences where soil excavation in planned, exterior

lead-based paint with the potential to recontaminate the yard after remediation should be controlled prior to the soil cleanup at the residence. Data collected from several sites across the Nation indicate the exterior lead-based paint has recontaminate yard soils that were remediated in as little as two to three years, recreating significant exposure risk to young children living in the homes.

3.2 Subsequent Actions/Non-Time-Critical or Remedial

Long-term cleanup actions, either non-time critical removals at non-NPL sites or remedial actions at NPL sites, should be initiated immediately at the conclusion of the time-critical actions. These follow-on cleanup activities shall include the actions described below.

Additional Yard Soil Sampling: At large sites where initial budgets were insufficient to characterize every property within the site, the remaining uncharacterized properties must be sampled until all yards potentially exceeding the site specific actions level are identified.

Yard Soil Removal: Continue with the cleanup of all residences above the site specific action level determined in the IEUBK Risk Assessment. Remediation of all day care facilities, parks, schools, and other green spaces where soil-lead exceeds the action level not previously addressed during the time-critical removal action should be given the highest priority.

Exterior Lead-Based Paint: Exterior lead-based paint with the potential to recontaminate the remediated residential yards should be controlled prior to the soil excavation at each residence requiring remediation.

Interior Dust: Interior dust attributed to exterior sources should be addressed after the yard soil cleanup at each residence. Once the soils have been remediated, a thorough one-time cleaning of all interior surfaces should be performed to mitigate the remaining exposure risk caused by dust in the home.

Source Control: Any sources that could potentially recontaminate residential yards should be controlled simultaneously to, or shortly after, the yard soil cleanup actions have been completed.

4. Soil Excavation Criteria

The following describes the requirements and procedures for excavation of yard soil from residential properties and HCIA.

Depth of Excavation: Generally, one foot of clean soil between the underlying contaminated soil and the yard surface in considered protective of young children playing in the yard. Therefore, excavation of one foot of soil is normally all that is required from a residential yard. Excavation depth should, however be based on the contaminant

concentration data generated during the sampling event. For example, if the depth sampling indicated the contamination is generally restricted to the upper six inches of soil, only a six-inch excavation would be required. Conversely, if the depth sampling indicates the contamination levels' exceeding the action extends to 18 inches, consideration should be given to excavating to 18 inches to removal all contamination. In any case, the excavation should generally not be beeper than 24 inches in the yard area.

Vegetable garden areas must be treated differently than the general yard area. Studies have shown that garden vegetables will uptake metals (primarily lead and cadmium) through roots to a depth of up to 24 inches. Therefore, existing gardens where soils exceed 500 ppm lead and/or 25 ppm cadmium must be excavated to a depth of at least 24 inches or until the soil concentration is less than 500 ppm lead and 25 ppm cadmium. In communities where vegetable gardens are extremely prevalent, consideration must be given to excavating the general yard soil to 24 inches or less than 500 ppm lead and 25 ppm cadmium.

Bottom of Excavation Barriers: EPA has received a health consult from ATSDR stating that 1,200 ppm lead or less below 12 inches of clean soil is considers protective without the installation of a barrier on the underlying soil. A barrier, heavy plastic construction or snow fencing, shall be placed in the bottom of the excavation to cover areas of the yard where soils are left above 1,200 ppm lead. Sampling must be conducted in the bottom of the excavation to determine the need for barrier installation prior to backfilling with clean soil. However, only XRF sampling results are needed for making this decision. No laboratory confirmatory samples should be collected from the bottom of the excavation.

Drip Zone: The drip zone soil in yards of older homes where exterior lead-based paint was applied generally will contain soils with significantly higher lead concentrations than the rest of the yard. It is not uncommon in these cases for parts of the yard to be less than the site specific action level for the site while the drip zone soil greatly exceeds. Therefore, to ensure the overall protectiveness of the remedy for each individual property addressed during the cleanup action, all portions of the drip zone that exceeds the action level should be excavated and removed. For example, the quadrant sampling data for a yard might show the only one quadrant in the back yard of the property exceeds the action level, but that the drip zone exceeds around the entire perimeter of the house. In this case, soil must be excavated from the back yard quadrant and the entire drip zone surrounding the house.

5. Source Control

Any sources that could potentially recontaminate residential yards within a short time-frame should be remediated prior to conducting the yard soil cleanup actions. Typical sources that would require remediation are mine waste piles located near enough to residences that yards would receive run off or wind blown dust, or smelter emissions containing high enough concentrations of metals that when deposited of yards through

fallout would recontaminate the yard within a matter of years. These types of recontaminations sources must be brought under control prior to remediation of residential yards.

F. RECORDS MANAGEMENT

Documentation of environmental data collection and analysis procedures (i.e. laboratory documentation, field log book, photo documentation, chain-of-custody) should be completed and managed using the requirements specified in the Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites.

G. QUALITY ASSURANCE AND QUALITY CONTROL

The Superfund Division Director in EPA Region 7 has the responsibility for oversight and assessment of this Regional protocol. This responsibility can be delegated to the Superfund Deputy Division Director and to Branch Managers within the Superfund Division.

H. REFERENCES

American Society for Testing and Materials (ASTM). 1998. *Standard Test Method for Particle-Size Analysis of Soils*. D 422-63. 1998.

U.S. Environmental Protection Agency (EPA). *Superfund Lead-Contaminated Residential Sites Handbook*. OSWER 9285.7-50. August. U.S. Environmental Protection Agency. Office of Emergency and Remedial Response. 2003.

U.S. Environmental Protection Agency (EPA). *Short Sheet: TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites*. April. OSWER Publication 9285.7-38. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. EPA Publication EPA/540-F-00-010. 2000.

U.S. Department of Housing and Urban Development (HUD). *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing*. June. 1995.

U.S. Environmental Protection Agency (EPA). Region 7. *Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites.* April. 2007.

STANDARD OPERATING PROCEDURE

4230.19B

Soil Sampling at Lead-Contaminated Residential Sites

March 31, 2012

Ronald King
SUPR/ERNB

APPROVED:

A. Mark Rooks — RPM Peer Review — Date 4/24/12

OSC Peer Review — Date 4/30/2012

Risk Assessor Peer Review — Date 5/2/12

Manager, Special Emphasis Remedial Branch — Date 8/8/12

Manager, Emergency Response and Removal North Branch — Date 5/8/12

Manager, Emergency Response and Removal South Branch — Date 8/9/12

Dianne Harris — Independent QA Reviewer — Date 05/22/2012

Recertified:

Branch/Name					
Date					

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A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures for the collection of representative surface soil samples at lead-contaminated residential sites as described in the Superfund Lead-Contaminated Residential Sites Handbook (Handbook, 2003). The sampling depths are specific to investigations for this type of site. Analysis of soil samples may determine whether concentrations of specific pollutants (e.g., lead, barium, cadmium, cobalt, copper, mercury, nickel and zinc) exceed established action levels, or if the concentrations of pollutants present a risk to public health, welfare, or the environment.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriate site report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (EPA) endorsement or recommendation for use.

B. SUMMARY OF METHOD

Soil samples may be collected using a variety of methods and equipment depending on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Surface soils may be easily sampled using a spade, trowel, and scoop.

The major category of sites where sampling will be performed includes, but is not limited to active/former lead mining, milling and smelter sites, areas impacted by mining, milling, and smelter activities, mining depositories, transportation routes from mining, milling and smelter sites and the use of mining wastes in public and residential areas.

C. DEFINITIONS

Residential properties: As defined in the Handbook, residential properties are any areas with high accessibility to sensitive populations, and include properties containing single- and multi-family dwellings, apartment complexes, vacant lots in residential areas, schools, day-care centers, community centers, playgrounds, parks, green ways, and any other areas where children may be exposed to site-related contaminated media.

X-Ray Fluorescence (XRF) spectrometer: An instrument used to resolve radiation into spectra to determine measurements. Will be used to analyze soils for metals contamination as described in the Instruction Manual for the XRF spectrometer.

Integrated Exposure Uptake Biokinetic Model (IEUBK) – Predicts blood-lead concentrations (PbBs) for an individual child, or group of similarly exposed children (under 84 months years of age), who are exposed to lead in the environment.

D. HEALTH AND SAFETY WARNINGS

Proper health and safety procedures must be observed during the investigation at all times. The Occupational Safety and Health Administration (OSHA) regulation for Hazardous Waste Operations and Emergency Response (HAZWOPER), specified in 29 CFR 1910.120(b)(4), requires a site-specific Health and Safety Plan (HASP) for each site where workers are engaged in handling/operations involving hazardous waste. In compliance with this regulation, all responding Region 7 personnel and their designated representatives are covered by a site-specific HASP developed to address the health and safety hazards, physical and chemical, which may be encountered at each site. The HASP also identifies procedures for protecting employees while on the site.

E. CAUTIONS

This section is not applicable to this SOP.

F. INTERFERENCES

This section is not applicable to this SOP.

G. PERSONNEL QUALIFICATIONS

All field personnel are required to take the 40-hour health and safety training course (as per 29 CFR 1910.120(b)(4)) and regular refresher courses prior to engaging in any field data collection activities.

H. EQUIPMENT AND SUPPLIES

Equipment and supplies used in the field to perform surface soil sampling may include but are not limited to:

- Maps/plot plan
- Safety equipment, as specified in the site-specific Health and Safety Plan
- Survey equipment or global positioning system (GPS) to locate sampling points
- Tape measure
- Survey stakes or flags
- Camera and film
- Stainless steel, plastic, or other appropriate homogenization bucket, bowl or pan
- Appropriate size sample containers
- Ziplock plastic bags
- Logbook
- Labels
- Chain of Custody records and custody seals

- Shipping container
- Field data sheets and sample labels
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel(s)
- Continuous flight (screw) auger
- Bucket auger
- Post hole auger
- Extension rods
- T-handle
- Sampling trier
- Thin wall tube sampler
- Split spoons
- Vehimeyer soil sampler outfit
  - Tubes
  - Points
  - Drive head
  - Drop hammer
  - Puller jack and grip
- Shaker sieve #10
- Shaker sieve (initially 250 micron #60 for risk assessment)
- X-Ray Fluorescence (XRF) spectrometer

I. PROCEDURAL STEPS

Soil screening activities will be conducted in accordance with the guidelines established in the Handbook.

1. PREPARATION

- Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.
- Obtain necessary sampling and monitoring equipment.
- Decontaminate or pre-clean equipment, and ensure that it is in working order.
- Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate.
- Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.
- Use stakes, flagging, or buoys to identify and mark all sampling locations.

Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location.

2. SAMPLING STRATEGY

The Handbook provides the sampling strategy when sampling residential properties. The sampling strategy is specific to the following categories:

- Residential yards;
- Drip zones;
- Play areas, gardens, and driveways;
- Potable water, lead-based paint, and interior dust; and
- Backfill and waste soil.

Soil sampling will be conducted in accordance with the guidelines established in the Handbook.

3. SAMPLING METHOD

3.1 Sample Collection

The Handbook describes the sampling depth when sampling residential properties. The following has been taken from this document.

Composite samples should consist of discrete aliquots of equal amounts of soil. The soil from each aliquot should be collected into one clean container, such as a stainless steel bowl or plastic bag, and thoroughly mixed. After mixing, the sample can then be analyzed by XRF spectrometer or sent to the laboratory. Follow EPA Method 6200 when using a XRF spectrometer. Remaining sample volume can then be disposed in the general location from where it was collected, or archived, depending on the requirements of the project. In some cases material other than grass and/or soil will be encountered at a sample location, e.g., wood chips and sand are often found in recreation areas of day-care and school playgrounds. Samples of the soil below the cover material should be collected.

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, spoons, and scoops. Surface material is removed to the required depth and a stainless steel or plastic scoop is then used to collect the sample.

This method can be used in most soil types but is limited to sampling at or near the ground surface. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A flat, pointed mason trowel to cut a block of the desired soil is helpful when undisturbed samples are required. Tools plated with chrome or

other materials should not be used. Plating is particularly common with garden implements such as potting trowels.

3.2 Sample Depth

The Handbook describes the sampling depth when sampling residential properties. Collection of samples from specified depth intervals serves two primary purposes: risk assessment and remedial decision-making. The following has been taken from this document.

3.2.1 Surface Soil Sampling For Risk Assessment Decision Making

With respect to risk assessment, the top inch of soil best represents current exposure to contaminants and is the source of data typically used in the IEUBK model to represent exposure from soil. This sampling should be done at all properties and will be used to determine whether a property exceeds the cleanup criteria and qualifies for response actions.

3.2.1.1 Surface Soil Sampling

A five-point composite surface soil samples should be collected from any portion within the 0- to 1-inch depth interval for human health risk assessment purposes. The samples should be collected using the procedure described in Section 3.1. If a measuring device is not used to determine the 1-inch depth, then the spoon or sampling device should sample the upper portion of the 0- to 1-inch interval to avoid going below the 1-inch depth.

3.2.1.2 Sampling to Define the Depth Profile

The sampling design discussed below is based on the assumption that a minimum of 12-inch soil cover is adequate.

Initial sampling for lead contamination in residential soils should also be conducted to a depth of at least 18 inches, but does not need to exceed 24 inches to define the vertical extent of contamination for cleanup purposes. Composite samples should be collected at 6 inch depth intervals, i.e., 0-6 inches, 6-12 inches, 12-18 inches, and 18-24 inches. Additional sampling may be required at lead sites when contamination is associated with coarse-grained material. Stone-sized material, such as tailings and crushed battery casings, will, over time, migrate upward through the soil via freeze/thaw effects. At such sites, composite sampling should be conducted at 6-inch intervals to the approximate maximum frost depth. In all cases, composites should consist of aliquots collected from the same depth interval.

In site-specific situations, deeper sampling may be conducted to determine the total vertical extent of contamination for groundwater issues or institutional controls (ICs), and to determine if complete removal of contaminated soil is possible. Depth

sampling should be conducted until the vertical extent of contamination has been adequately defined, but does not need to be conducted on every property.

3.2.1.3 Sampling to Determine if a Cleanup has been Completed

The Handbook does not distinguish between sampling to assess a property for cleanup and sampling to determine if cleanup has been completed. However confirming that all contaminated soil has been removed before backfilling involves different sampling procedures. Specifically, aliquots are collected from zero to six inch intervals instead of the zero to one inch intervals used when sampling to assess a property for cleanup.

Use of a soil sampling probe will help with sample collection. It is difficult to push the probe exactly six inches below the ground surface. It is usually necessary to withdraw the probe from the ground and collect only the top six inches of the soil column for the composite sample.

3.3 Sample Preparation

The Handbook describes the sampling preparation when sampling residential properties. The following has been taken from this document.

Composite samples should consist of discrete aliquots of equal amounts of soil. The soil from each aliquot should be collected into one clean container, such as a stainless steel bowl or plastic bag, and thoroughly mixed.

Samples collected from all depth intervals should be dried, sieved with a No. 10 sieve (2 mm), and homogenized. Samples should not be ground prior to sieving, as this changes the physical structure of the soil and may bias the analytical results.

For those soil samples that are collected for risk assessment purposes, the sample will also be processed through a No. 60 sieve (250 μm) to obtain the fine fraction. The EPA Technical Review Workgroup (TRW) and American Society for Testing and Materials (ASTM) have issued guidance on sieving (ASTM, 1998; EPA, 2000). To reduce sampling costs, it may be desirable to develop a correlation between sieved and unsieved data, to eliminate the need to sieve all samples. The correlation can be used to predict sieved results from unsieved samples. The EPA TRW guidance addresses appropriate sieve size (No. 60) and a method for predicting the concentration in the fine fraction using concentrations measured in unsieved samples. A portion of each homogenized sample from each sampling area will be screened for lead using XRF spectrometer or submitted for laboratory analysis.

3.4 Sample Analysis

The Handbook describes the sampling analysis when sampling residential properties. The 4220.03A SOP should also be consulted for decision making for using the XRF spectrometer.

J. DATA AND RECORDS MANAGEMENT

Documentation of environmental data collection and analysis procedures (i.e. laboratory documentation, field logbook, photo documentation, chain-of-custody) should be completed and managed using the requirements specified in the Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites.

K. QUALITY ASSURANCE AND QUALITY CONTROL

Care should be taken to follow the Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP) associated with any sampling activity. The following QA procedures should be included in the FSP and QAPP:

1. All data must be documented on field data sheets or within site logbooks.

2. XRF spectrometers should be operated according to the SW-846 *Method 6200 - Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. Accuracy checks are performed using certified prepared standards daily. Record these accuracy checks in the field logbook. The following information is recorded.

- Equipment identification (name) and control number.
- Date of accuracy check.
- Activity performed on instrument.
- Adjustments made and accuracy of equipment before and following accuracy check (where applicable).
- Record of equipment failure.
- Identification of person performing accuracy.

L. REFERENCES

American Society for Testing and Materials (ASTM). 1998. *Standard Test Method for Particle-Size Analysis of Soils*. D 422-63.

U.S. Environmental Protection Agency (EPA). 2000. *Short Sheet: TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites*. April. OSWER Publication 9285.7-38. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. EPA Publication EPA/540-F-00-010.

U.S. Environmental Protection Agency (EPA). 2003. *Superfund Lead-Contaminated Residential Sites Handbook*. OSWER 9285.7-50. August. U.S. Environmental Protection Agency. Office of Emergency and Remedial Response.

U.S. Department of Housing and Urban Development (HUD). 1995. *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing*. June.

U.S. Environmental Protection Agency (EPA). Region 7. 2007. *Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites.* July.

Method 6200 - Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment from Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, also known as SW-846.

STANDARD OPERATING PROCEDURE

No. 2420.5D

IDENTIFICATION, DOCUMENTATION, AND TRACKING OF SAMPLES

December 16, 2003

by

Harry Kimball

ENSV/RLAB/CATS

APPROVED:

Peer Reviewer — Date: 12/18/03

Manager, Regional Laboratory — Date: 19 Dec 03

Independent QA Reviewer — Date: 12/19/03

Recertified

Reviewer					
Date					

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A. PURPOSE AND APPLICABILITY

The purpose of this standard operating procedure (SOP) is to establish uniform procedures for assigning sample numbers, labeling sample containers, documenting the sample collection process, and for tracking samples.

The collection of samples is an essential step in the process for obtaining information on a variety of environmentally-related conditions and situations. Because the analytical results of samples are used extensively to support regulatory decisions, statutory actions, environmental and health assessments, and litigation proceedings, a critical component of the sample collection process is the proper identification, documentation, and tracking of each sample collected.

The procedures outlined herein are applicable to all samples received by the Region 7 Laboratory (RLAB) for analysis (either in-house analysis or out-source contract lab analysis) and to laboratory-generated quality control (QC) samples. The Regional Sample Control Coordinator (RSCC), or their surrogate, shall ensure, at the time of sample receipt, that samples received by RLAB conform to the identification and documentation requirements of this SOP. This SOP should be provided to all individuals (EPA, state, and tribal staff, plus their contractors) collecting samples for delivery to RLAB to facilitate compliance with these procedures.

B. SUMMARY OF PROCEDURE

1. The identification and documentation of each sample is required in order to provide tangible evidence that shows the data resulting from sample analysis is linked directly to the sample collected. The basic mechanism used to establish this critical link between samples collected and analytical data is the assignment of a unique sample identifier to each sample collected, with supporting written information to document the sampling process. In addition to providing the means for establishing the relationship between samples and analytical results, the assignment of unique sample identifiers provides a means for tracking samples through the analytical data generation process.

2. Sample identification is achieved by labeling each field collected sample with a unique sample identifier. Samples contained in multiple sample containers will bear the same unique sample identifier on each container, plus, each container will be uniquely identified (usually by analysis). Quality control is an integral part of the process of obtaining reliable information about environmental samples, therefore, field and laboratory quality control samples will be uniquely identified in an appropriate and consistent manner.

3. Sample documentation is accomplished by recording the appropriate information about the sample on a field sheet which bears the sample's unique sample identifier. If samples are delivered to RLAB with sample identifiers that are not consistent with the unique sample identifiers described in this SOP, the RSCC will assign the requisite unique sample identifiers and record the original sample identifier in the LIMS "External Sample Number" field. Laboratory QC samples are documented on the sample prep and/or analysis log.

4. Sample tracking is accomplished by using the Region 7 Laboratory Information Management System (LIMS). The LIMS is used to identify and track the status of all samples analyzed by the EPA Region 7 Laboratory and its contractors. The current LIMS is a product called R7LIMS. R7LIMS and any future LIMS products will follow the sample identification scheme defined in this SOP. Additionally, the LIMS can generate field sheets and tags (sample labels) to facilitate identification and documentation of field collected samples (see SOP 2420.13, "RLAB Procedures for Preparation of Field Sheets and Tags"). The physical location of samples is tracked by chain-of-custody procedures.

5. Because the identification and documentation of samples establishes the foundation for substantiating reported analytical data, it is important that the individuals who collect and/or generate samples follow the procedures contained in this SOP. The procedures contained in SOP No. 2420.4, "Field Chain-of-Custody for Environmental Samples," should be used in conjunction with this SOP to provide complete field sample documentation.

C. DEFINITIONS

The following definitions of commonly-used terms relating to types of samples and sampled matrices are provided for clarification in the sample identification process:

1. Sample. The word 'sample' is an often overworked term. It can refer to a sample collected in the field, a portion of a field sample that has been spiked with additional analytes (matrix spike sample), or a sample generated entirely within the laboratory, such as a method blank. The term 'sample' most often refers to a Field Sample that is of one matrix collected from a specific point (or area if spatially composited) at a specific time (or period of time if temporally composited). A sample may be divided into several different containers, each for a different type of analysis and possibly requiring different methods of preservation (see SOP 2420.6, "Sample Container Selection, Preservation and Holding Times"). It is common for all of these containers to be collectively referred to as being a (one) sample and for all of them to bear the same unique sample identifier.

2. Field Sample. A representative portion of an environmental matrix (e.g. air, soil, water, etc.) collected from a specific location at a specific time to obtain information regarding environmental conditions and/or effects, process operations and material contents. Field Samples are actual portions of a matrix collected to determine its physical, chemical, or biological constituents and are distinguished from samples used for quality control (QC) purposes. Although QC samples collected in the field are in a sense field samples, the term Field Sample is used to denote a non-QC sample and is sometimes referred to as a "real" or "regular" Field Sample. Field Samples include those collected to evaluate background conditions and are categorized as grab, composite or continuous samples.

   a. Grab Sample. A discrete portion of a matrix collected at a specific location at one instance in time (this period of time is typically defined as not exceeding 15 minutes to allow adequate time for sample collection under most field situations). This type of sample is representative of the environmental condition at the time of collection. This type of sample is commonly used for in-situ determinations and for obtaining information on constituents that require special handling or may be lost if sampled in another manner.

   b. Composite Sample. A portion of a matrix consisting of a mixture of two or more discrete portions (grab samples) collected from a specific location over a period of time or from a specific area (multiple locations) at one time or over a period of time. This type of sample is a representative average of the environmental condition for a definable area and/or period of time. This type of sample is commonly used for assessing environmental conditions.

   c. Continuous Sample. As the name implies, it is a representative portion of a matrix collected in an uninterrupted manner for a period of time. This type of sample is normally associated with in-situ determinations and is, therefore, not usually collected for submittal to a laboratory for analysis. Continuous samples are most commonly used for collecting data of air and water media; e.g., flow, pH, temperature, etc.

3. Split Sample. As the name implies, it is a sample that is separated or split from the total amount of material sampled and sent to a different laboratory for analysis. Soil matrix samples are homogenized then split to ensure uniformity. The Split samples are used to independently verify laboratory analysis.

4. Extract. An extract is the result of the extraction process. The sample extract is labeled by extraction personnel.

5. Digestate. A digestate is the result of the digestion process. The sample digestate is labeled by digestion personnel.

6. Quality Control Sample. Prepared in the laboratory, in the field, or combination thereof, a QC sample is incorporated into sample collection and/or analysis activities as a means of evaluating the quality of analytical results obtained from Field Samples. This type of sample may be a field-collected sample (e.g. duplicate sample) or a laboratory-generated sample, depending on its intended purpose, to evaluate and/or substantiate analytical results. Additional information on the use of QC samples for calculating data quality may be found in SOP No. 2410.15, "Estimating and Documenting Data Quality". The following types of QC samples are commonly encountered in sampling events and should be sufficient to categorize most QC samples:

   a. Duplicate Sample. It is recognized that there are several interpretations of this term. For the purpose of calculating data quality, there are essentially two types of duplicate samples: field and laboratory, as described below.

      (1) Field duplicate samples refer to two Field Samples collected simultaneously from the same location(s) under identical conditions. A duplicate grab sample consists of collecting two Field Samples at the same location and time. A duplicate composite sample consists of two Field Samples containing multiple grab samples each collected at the same location and time. If automatic samplers are used to collect composite samples, the collection of duplicate composite samples would require two automatic samplers to be collocated and set to collect the individual portions or aliquots at the same times. The dividing (also referred to as "splitting") of a single sample into two portions will be considered field duplicate samples in those situations where the preferred method of simultaneous collection cannot be met due to field conditions (e.g. the media being sampled is non-homogeneous like some soils, gravel, etc.).

      (2) Laboratory duplicate samples refer to equivalent aliquots taken from a single sample received by a laboratory for analysis as unique samples. The process of obtaining the duplicate aliquots should be preceded by ensuring the sample is well mixed.

   b. Blank Sample. A sample that is presumed to be free of contamination from constituents of concern and is designed to detect contamination due to the sampling and/or analysis process (collection, preservation, handling, sampling environment, extraction, analysis, etc.).

(1) Field Blank. Includes all blank samples which are prepared in or enter the field environment and include trip blanks, equipment blanks, bottle or container blanks, reagent or preservative blanks and tubing blanks. Ideally, a field blank for most analytical parameters should be exposed to the sampling, preservation and handling process used to collect the physical samples, but this may not always be possible (e.g. the field blanks for volatile organics are only transported unopened to and from the sampling environment). The type of field blank should be identified, as well as the group of Field Samples with which it is associated, in the appropriate sample documentation.

(a) Trip Blank. It is a sample that is presumed to be free of contamination from constituents of concern, and is carried into the field and returned while being exposed to the same field conditions which the sample containers experience during the sample shipping process.

(b) Tubing/Equipment Blank. It is a sample free from constituents of concern (normally deionized water that is distilled) and is pumped through or otherwise introduced into the sampling equipment. The process results in exposure of the sample to any constituents of concern which might be contained in or on the surfaces of the sampling equipment.

(c) Preservation Reagent Blank. It is a sample which is originally free from constituents of concern (normally distilled deionized water) and to which the preservative (acid or other chemical) is added in the same concentration and quantity as normally added to a sample. The purpose is to determine if any contaminants of concern exist in the preservative used.

(d) Container Blank. A sample originally free from constituents of concern (normally distilled deionized water) which is introduced into randomly chosen containers at the time of sampling. The purpose of this blank is to determine the existence of contaminants of concern in the sampling containers.

(2) Method Blank. A laboratory QC sample used to assess the level of contamination in the analytical system. A method blank is,

typically, a portion of a clean matrix that is taken through the entire sample preparation and analysis process.

c. Laboratory QC Sample. A variety of QC samples are used by an analytical laboratory for internal QC purposes. For the purpose of sample identification, all such samples prepared by the laboratory for internal use are classified under this category. Commonly used laboratory QC samples include lab duplicate samples, method blanks, lab control samples, matrix spikes, and lab fortified blanks.

d. Performance Evaluation Sample. A sample that contains a known amount of a chemical constituent or parameter and is introduced for analysis to assess the accuracy of the analytical method. The actual content of the PE sample, either in regard to specific constituents and/or concentrations of constituents, is normally unknown to the receiving analytical laboratory.

e. Performance Testing Sample. Similar to a performance evaluation sample except that it is provided by a NELAC (National Environmental Laboratory Accreditation Conference) certified PT sample provider. Results of the analysis of these samples are used for NELAC accreditation purposes.

f. Some additional Field Samples may be thought of as QC samples due to the location or method of sample collection. These are labeled the same as, and analyzed the same as, other Field Samples.

(1) Rinsate Sample. This type of sample is used to evaluate the effectiveness of field decontamination procedures for sampling equipment. The sample is obtained by collecting the rinse water that is poured over the sampling equipment after decontamination has been completed (the water is normally distilled ionized water prepared in the laboratory and carried to the field).

(2) Background Sample. In some investigations, samples are collected to determine what is representative of the environment for constituents of concern. These samples, normally called background samples, are Field Samples which are collected off-site or upstream of an area that is affected by a contaminant of concern, but are not expected to contain any or significant amounts of the contaminant of concern.

7. Matrix. The matrix (also known as 'media') refers to the substance from which the sample was obtained and/or of which the sample consists. Since the sampled matrix has a direct bearing on how a sample is preserved and on the selection of

the method to analyze the sample, the identification of the matrix is an important aspect of sample documentation.

a. RLAB Matrix. The RLAB matrix is the matrix name used by RLAB to identify the matrix of the sample. It is the matrix used in the LIMS and in the RLAB Methods.

(1) Air. All samples collected to evaluate or analyze the chemical and physical contents of the air, both indoor and outdoor. The resulting sample may be in different forms depending on the method of collection (e.g. Tenex tube, canister, PUF, etc.).

(2) Solid. All samples obtained of soils, sediments, sludge, dust, and any other solid material.

(3) Tissue. All samples obtained of living organisms; e.g., plants or vegetation, fish, animals, etc., either whole or portions thereof.

(4) Waste. All samples obtained of media that do not logically fit under one of the other specifically defined matrices or contain exceedingly high concentrations of analytes. (Previously referred to as "Hazardous/Other".) Examples of these type samples are wipe samples, drum samples, non-aqueous liquid samples, product or formulation samples and mixed media samples.

(5) Water. All samples obtained of aqueous liquid, e.g., wastewater, surface water, drinking water, groundwater, etc.

b. NELAC Matrix. NELAC has its own list of Quality System Matrices. These matrices are referenced in the RLAB Methods, but are not used in the LIMS or for sample definition/identification.

(1) Aqueous. Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

(2) Drinking Water. Any aqueous sample that has been designated a potable or potential potable water source.

(3) Saline/Estuarine. Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

(4) Non-aqueous Liquid. Any organic liquid with <15% settleable solids.

(5) Biological Tissue. Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

(6) Solids. Includes soils, sediments, sludges and other matrices with >15% settleable solids.

(7) Chemical Waste. A product or by-product of an industrial process that results in a matrix not previously defined.

(8) Air and Emissions. Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

D. PERSONNEL QUALIFICATIONS

Personnel collecting and/or delivering samples to RLAB should have a basic knowledge and understanding of RLAB sample management procedures including chain-of-custody (SOP 2420.4). RLAB personnel receiving samples must be knowledgeable of the sample log-in process (SOP 2420.1, "Sample Receipt and Log-in"). Personnel defining samples in the LIMS must be familiar with using the LIMS (SOP 2410.20, "R7LIMS Functions and Security") and have an R7LIMS account.

E. SAMPLE IDENTIFICATION

1. Each sample is identified by a unique sample identifier which is assigned to it.

   a. This identifier is used to distinguish an individual sample from all other samples and is used on all documentation relating to collection, handling, analysis and reporting the analytical results of an individual sample.

   b. Since a sample is normally analyzed for a number of different chemical constituents or parameters that require different sample containers and preservation techniques, the same unique sample identifier will be assigned to each portion of the original sample split among individual sample containers. For example, if a sample is split among three individual sample containers in order to properly preserve each portion for the specific parameter or group of parameters to be analyzed, each of the

individual sample containers would be identified by the same unique sample identifier.

2. The unique sample identifier consists of three parts: the Analytical Services Request Number (ASR Number), Sample Number, and Quality Control Code (QC Code). These are frequently written together, separated by hyphens. The unique sample identifier is sometimes (confusingly) simply referred to as the sample number.

   a. ASR Number - This is the number automatically assigned to an ASR at the time it is defined in the LIMS. Each ASR has its own unique number.

   b. Sample Number - This number is assigned by the responsible Project Manager (or their designee) for each field sample collected for an ASR.

   c. QC Code - This two or three character alpha code is used to identify the nature of the sample for QC purposes. Field personnel will normally only use the following codes to identify field collected samples:

   __ = Field Sample (two underscore characters)
   FD = Field Duplicate
   FB = Field Blank
   FS = Field Spike
   FSD = Field Spike Duplicate

   Laboratory personnel will use the following codes to identify laboratory QC samples:

   MB = Method Blank
   LD = Laboratory Duplicate
   MS = Matrix Spike
   MSD = Matrix Spike Duplicate
   LCS = Laboratory Control Sample
   LFB = Laboratory Fortified Blank
   PE = Performance Evaluation sample
   PT = NELAC Performance Testing sample

3. The following examples are provided to illustrate some unique sample identifiers:

   26-1-__ - Field Sample number 1 for ASR Number 26
   26-1-FD - Field Duplicate of Field Sample above
   26-2-FB - Field Blank submitted for same ASR Number
   87-5-__ - Field Sample number 5 for ASR Number 87

87-5-MS - Matrix spike of Field Sample above
87-900-LCS - Lab Control Sample number 900 for ASR Number 87

4. Some quality control samples have meaning only when referenced to another sample (i.e. QC Codes of FD, FS, FSD, LD, MS, MSD). To facilitate the identification of the referenced sample, the LIMS has two fields for use with these QC samples: Ref Sample Number and Ref QC Code. Rules for determining the Sample Number, Ref Sample Number, and Ref QC Code for these QC samples are given below.

   a. The QC sample and the referenced sample (the sample that the QC sample is a spike or duplicate of) must have the same ASR Number and Matrix.

   b. Field QC samples (FD, FS, FSD) will be assigned the same Sample Number as the original Field Sample (__) that they are a duplicate or spike of. The Ref Sample Number, and Ref QC Code are automatically assigned by the LIMS and can not be edited by the user.

   c. Lab QC samples (LD, MS, MSD) that are a duplicate or spike of a Field Sample or Performance Testing sample (__, PT) will be assigned the same Sample Number as the original Field Sample or Performance Testing sample that they are a duplicate or spike of. By default, the Ref Sample Number will be set to the Sample Number and the Ref QC Code will be set to "__" by the LIMS. If the sample being spiked or duplicated is a Performance Testing sample, a Ref QC Code of "PT" will need to be manually entered into the LIMS. Note that it is not appropriate for a Field Sample and a Performance Testing sample to have the same Sample Number.

   d. Lab QC samples (LD, MS, MSD) that are a duplicate or spike of any other field collected sample (QC Code of FB, FD, FS, FSD) will be assigned a different Sample Number than the original sample that they are a duplicate or spike of. The Ref Sample Number and Ref QC Code will need to be manually entered into the LIMS. Although not a requirement, it is suggested that a Sample Number in the "800" range be used for the lab QC sample.

   e. MSD samples must have the same Sample Number, Ref Sample Number, and Ref QC Code as their associated MS sample. The MS sample must be defined in the LIMS before the MSD sample can be defined.

5. The following rules are provided for further clarification of the unique sample identifier assignment process:

a. Each sample collected of a specific media will have a unique sample identifier. For example, if two samples are collected at the same location and time, but are of two different media (e.g. air and solid, or water and tissue), the sample of each specific media will be considered a separate sample. Each sample will be assigned a separate sample number.

b. In-situ samples collected for instantaneous field determinations (e.g. pH, temperature, specific conductance, dissolved oxygen, residual chlorine) in connection with the collection of samples for submission to a laboratory for analysis will be identified by sample identification numbers. Results of field determinations are recorded on field sheets associated with the sample collected for laboratory analysis. The sample identification number of the sample used for the field determination will normally be the same as the sample identification number of the sample submitted for analysis.

c. Continuous samples do not require the assignment of sample identification numbers, but do require specific written documentation to record sampling locations, and times of sampling and readings. Since many continuous monitors provide strip charts and/or printouts of readings, this documentation should be kept to supplement other written documentation.

d. Even though samples for some analyses, such as those for volatile organics, are always collected in two or more containers, they are considered to be a single sample. Additionally, if multiple analyses are to be analyzed for (such as metals, pesticides and VOAs), separate containers will be needed for each analysis. These containers are collectively considered to be one sample and will have the same unique sample identifier.

e. Sample extracts are labeled by the person performing the extraction of the sample. The sample extract container is labeled by hand-copying the sample label's information onto a smaller sample extract label. The sample extract label must identify the extraction solvent. Transcription errors are prevented by double checking the sample extract label prior to affixing the sample extract label to the sample extract container. The sample extract label is then affixed to the sample extract container.

f. Digestates are labeled by the person performing the digestion of the sample. The sample digestate container is labeled by hand-copying the sample label's information onto a blank label. The sample digestate label must identify the requested analysis. Transcription errors are prevented by double checking the sample digestate label prior to affixing the sample

digestate label to the sample digestate container. The sample digestate label is then affixed to the sample digestate container.

g. As a general rule-of-thumb, Field Blanks that are associated with a group of samples will have their own Sample Number. Field Blanks that are associated with just one Field Sample (e.g. a separate Field Blank for each Field Sample) may have, but are not required to have, the same Sample Number as the Field Sample that it is associated with.

h. It is common practice for some laboratory QC samples (MB, LCS, LFB) to be assigned a Sample Number in the "900" range. This is not a requirement for these samples (any number may be used), however, it is a desirable practice as it helps avoid confusion by keeping these QC samples "numerically segregated" from Field Samples. For sampling events involving a large number of Field Samples, running into the 900 range, it may be desirable to number these QC samples in the 1500, 2000, or other appropriate range.

6. All samples submitted for analysis will have a sample label affixed to each sample container.

a. Sample labels currently in use are computer generated, therefore, minimal or no entries are required. Any entries made on the sample labels will be accomplished using indelible ink.

b. With the exception of volatile samples and samples packed inside a paint can for shipping, only one sample label is needed for each sample container. Since volatile and over-packed samples consist of more than one container, multiple labels are required so that each container (including the outside container) can be labeled.

NOTE: Since some of the computer-generated sample labels are susceptible to deterioration from water, clear plastic tape should be placed over these sample labels if they will come into contact with water (including ice) during storage, transport and/or shipment. Some computer-generated sample labels are water resistant; these labels will not require tape protection.

c. Each sample container must be uniquely identified by the sample label. Where there is only one container for an analysis (such as Metals in Water by ICP), the container is uniquely identified by the unique sample identifier (ASR Number, Sample Number, and QC Code) and the analysis abbreviation (such as Met W). Where there is more than one container for an analysis (such as VOCs in Water by GC/MS), the containers are

uniquely identified by the unique sample identifier (ASR Number, Sample Number, and QC Code), the analysis abbreviation (such as VOA W), and a sequential container number (1, 2, 3, etc.). "Specific" sample container labels generated by the laboratory's LIMS are uniquely identified as described above. When samples are received by the laboratory bearing LIMS "Generic" labels, labels generated by the sampler, or hand-made labels, the necessary additional information should be added to the label or a second label should be placed on the container to uniquely identify it. It is the responsibility of the laboratory person receiving the samples (RSCC or their alternate) to ensure that each container is uniquely identified.

F. SAMPLE DOCUMENTATION

1. A field sheet is used to document the field sample collection process and contains pertinent information relative to the sample collected. (Laboratory QC samples are documented on the sample prep and/or analysis log as described in SOP 2410.10, "Analytical Data Submission Package Contents and Review". This section deals primarily with field collected samples.)

2. A field sheet will be completed for each sample collected and will be the official document that provides a permanent record of each sample collected. Since this document is the essential written component required to establish the relationship between the sample collected and the analytical results obtained, it will be controlled and will become a part of the official file on a sampling event.

3. Field sheets can be generated by the laboratory's LIMS, or alternate forms may be used. A field sheet should contain, at a minimum, the following information:

   a. Unique Sample Identifier - This may be recorded as three separate pieces of information (ASR Number, Sample Number and QC Code) or written as one entry (separated by hyphens).

   b. Matrix Sampled - The RLAB matrix as defined in section C.7.a.

   c. Project Information - This should include such things as the Project Manager, Project ID and description, city, state and other pertinent information.

   d. Location/Description - This short description should identify, to the satisfaction of the Project Manager, where the sample was collected. This is typically done by describing or naming the sample collection location.

e. Sample Collection Date/Time - For time-composited samples, the start date and time and end date and time are required. For grab samples only the start date and time are needed. Times should be recorded in the 24-hour format.

f. Analyses - An unambiguous list of the required laboratory analyses.

g. Field Measurements - Recorded along with the measurement units.

h. Comments - As appropriate.

i. Sampler - The name of the person(s) collecting the sample.

4. The Project Manager is responsible for ensuring that all field sheets are properly and accurately completed, and are safeguarded until they are delivered to RLAB.

5. The original completed field sheets for each sampling activity will be delivered to RLAB along with the samples to be analyzed. They will be maintained in the RLAB analytical support file for the specific ASR.

6. All entries on the field sheets will be legible and completed in indelible ink. Corrections to entries on field sheets should be accomplished by drawing a single line through the entry to be corrected, entering the correction above or adjacent to the lined-through entry and dating and initialing the correction.

7. In addition to the field sheet, another essential component of sample documentation is chain-of-custody. SOP 2420.4 describes the procedures for chain-of-custody of field collected samples being delivered to RLAB. SOP 2420.2, "Storage and Security of Environmental Samples" describes chain-of-custody procedures for within-lab sample transfers of routine samples. For samples that are connected with a criminal investigation, SOP 2420.10, "RLAB Procedures for Custody and Tracking of Samples and Analytical Data Files to be used as Evidence in Criminal Investigations," describes chain-of-custody documentation procedures for within-lab sample transfers.

G. SAMPLE TRACKING

1. The LIMS database system is used for tracking the status of samples and sample analyses through the analytical process and for tracking and reporting the results of sample analysis. Numerous reports are available from the LIMS and provide a variety of information pertaining to the samples and sample analyses. SOP 2410.20 and the LIMS online help provides more information on this.

2. Information relating to the status of samples submitted for analysis and the status of sample analyses may be obtained by the Project Manager from the LIMS or the RLAB Data Coordinator.

3. It is recognized that changes frequently occur in the field which result in changes to planned sampling activities. Since the LIMS system is used for logging in samples upon receipt, tracking, and ultimately reporting the results, it is essential that Project Managers ensure the entries contained in LIMS for specific sampling activities are accurate and complete (especially any field data and measurements). Discrepancies relating to numbers and types of samples and parameters requested for analysis must be corrected at the time of sample receipt by RLAB in accordance with SOP 2420.1.

4. SOP 2420.2 describes, for routine samples, the procedures for tracking the location of samples and sample containers within the laboratory. For samples that are connected with a criminal investigation, SOP 2420.10 describes the procedures used for tracking the location of samples and sample containers within the laboratory.

5. Unless otherwise requested, environmental samples will be properly disposed of in accordance with SOP 2420.9, "Sample Disposal", upon completion of the analysis and finalization of the analytical results.

H. QUALITY ASSURANCE AND QUALITY CONTROL

It is incumbent on all parties involved with sample collection, analysis, and management that these procedures be followed. Conformance with these procedures shall be evaluated during scheduled audits of RLAB operations as described in SOP 2430.5, "Quality Control Spot Checks of Regional Laboratory Data Packages", and SOP 2430.6, "Periodic Internal Program Review of the Region 7 Laboratory".

I. REFERENCES

1. Region 7 SOP 2410.10, Analytical Data Submission Package Contents and Review
2. Region 7 SOP 2410.15, Estimating and Documenting Data Quality
3. Region 7 SOP 2410.20, R7LIMS Functions and Security
4. Region 7 SOP 2420.1, Sample Receipt and Log-in
5. Region 7 SOP 2420.2, Storage and Security of Environmental Samples
6. Region 7 SOP 2420.4, Field Chain-of-Custody for Environmental Samples
7. Region 7 SOP 2420.6, Sample Container Selection, Preservation and Holding Times
8. Region 7 SOP 2420.9, Sample Disposal
9. Region 7 SOP 2420.10, RLAB Procedures for Custody and Tracking of Samples and Analytical Data Files to be used as Evidence in Criminal Investigations
10. Region 7 SOP 2420.13, RLAB Procedures for Preparation of Field Sheets and Tags
11. Region 7 SOP 2430.5, Quality Control Spot Checks of Regional Laboratory Data Packages
12. Region 7 SOP 2430.6, Periodic Internal Program Review of the Region 7 Laboratory

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed below for soil and sediment samples. Some common elements are not listed in this method because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). These light elements are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed below are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF. The following RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-0
Barium (Ba)	7440-39-3
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5

Analytes	CAS Registry No.
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

In addition, the following non-RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Calcium (Ca)	7440-70-2
Iron (Fe)	7439-89-6
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-93-7
Potassium (K)	7440-09-7
Rubidium (Rb)	7440-17-7
Strontium (Sr)	7440-24-6
Thorium (Th)	7440-29-1
Titanium (Ti)	7440-32-6
Zirconium (Zr)	7440-67-7

1.2 This method is a screening method to be used with confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)). This method's main strength is that it is a rapid field screening procedure. The method's lower limits of detection are typically above the toxicity characteristic regulatory level for most RCRA analytes. However, when the obtainable values for precision, accuracy, and laboratory-established sensitivity of this method meet project-specific data quality objectives (DQOs), FPXRF is a fast, powerful, cost effective technology for site characterization.

1.3 The method sensitivity or lower limit of detection depends on several factors, including the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. Example lower limits of detection for analytes of interest in environmental applications are shown in Table 1. These limits apply to a clean spiked matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (100 -600 second) count times. These sensitivity values are given for guidance only and may not always be achievable, since they will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of performance-based sensitivity is presented in Sec. 9.6.

1.4 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use and operation of an XRF instrument. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use either sealed radioisotope sources or x-ray tubes to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples. The three electron shells include the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α), beta (β), or gamma (γ) etc., which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than

the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.77 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments, specifically, in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF -- Field portable x-ray fluorescence.

3.2 MCA -- Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS -- Site-specific calibration standards.

3.4 FP -- Fundamental parameter.

3.5 ROI -- Region of interest.

3.6 SRM -- Standard reference material; a standard containing certified amounts of metals in soil or sediment.

3.7 eV -- Electron volt; a unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One, Chapter Three, and the manufacturer's instructions for other definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup (i.e., against the cup window), the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_β line of element Z-1 with the K_α line of element Z. This is called the K_α/K_β interference. Because the K_α:K_β intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_α and K_β energies are 4.95 and 5.43 keV, respectively, and the Cr K_α energy is 5.41 keV. The Fe K_α and K_β energies are 6.40 and 7.06 keV, respectively, and the Co K_α energy is 6.92 keV. The difference between the V K_β and Cr K_α energies is 20 eV, and the difference between the Fe K_β and the Co K_α energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_α/lead (Pb) L_α and sulfur (S) K_α/Pb M_α. In the As/Pb case, Pb can be measured from the Pb L_β line, and As can be measured from either the As K_α or the As $K_ß$ line; in this way the interference can be corrected. If the As K_β line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_α line. If the As K_α line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for As, regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator’s decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-

atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)).

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as Method 3050, or a total digestion procedure, such as Method 3052, is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project-specific data quality objectives (DQOs).

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method (see Table 8), the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r often exceeding 0.95, except for barium and chromium). The critical factor is that the digestion procedure and analytical reference method used should meet the DQOs of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Sec. 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10° F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The user is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

NOTE: No MSDS applies directly to the radiation-producing instrument because that is covered under the Nuclear Regulatory Commission (NRC) or applicable state regulations.

5.2 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals.

Licenses for radioactive materials are of two types, specifically: (1) a general license which is usually initiated by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) a specific license which is issued to named persons for the operation of radioactive instruments as required by local, state, or federal agencies. A copy of the radioactive material license (for specific licenses only) and leak tests should be present with the instrument at all times and available to local and national authorities upon request.

X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply, however, if the tube is properly positioned within the instrument, this is only a negligible risk. Any instrument (x-ray tube or radioisotope based) is capable of delivering an electric shock from the basic circuitry when the system is inappropriately opened.

5.3 Radiation monitoring equipment should be used with the handling and operation of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs or badges should be worn in the area of maximum exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for

use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

6.1 FPXRF spectrometer -- An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation sources -- FPXRF instruments use either a sealed radioisotope source or an x-ray tube to provide the excitation source. Many FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron Fe-55 (^{55}Fe), cadmium Cd-109 (^{109}Cd), americium Am-241 (^{241}Am), and curium Cm-244 (^{244}Cm). These sources may be contained in a probe along with a window and the detector; the probe may be connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. This is due to the ever increasing time required for the analysis rather than a decrease in instrument performance. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum necessary for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of

accelerating voltage is governed both by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material and by the instrument's ability to cool the x-ray tube. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample presentation device -- FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For FPXRF instruments operated in the intrusive mode, the probe may be rotated so that the window faces either upward or downward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors -- The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon Si(Li). The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -90 °C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 L. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2-270 eV; silicon pin diode-250 eV; Si(Li)–170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data processing units -- The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the

units or from PCs. Once the data–storage memory of an FPXRF unit is full or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

6.3 Polyethylene sample cups -- 31 to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film -- Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 µm thick.

6.5 Mortar and pestle -- Glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers -- Glass or plastic to store samples.

6.7 Sieves -- 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels -- For smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags -- Used for collection and homogenization of soil samples.

6.10 Drying oven -- Standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Pure element standards -- Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if designated for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.3 Site-specific calibration standards -- Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.3.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of 10 samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.3.2 Each sample should be oven-dried for 2 to 4 hr at a temperature of less than 150 °C. If mercury is to be analyzed, a separate sample portion should be dried at ambient temperature as heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be homogenized (see Sec. 7.3.3) and then a representative portion ground with a mortar and pestle or other mechanical means, prior to passing through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.3.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 g of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 g of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.4 Blank samples -- The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the established lower limit of detection. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.5 Standard reference materials -- Standard reference materials (SRMs) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. When these SRMs are unavailable, alternate standards may be used (e.g., NIST 2702).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, "Inorganic Analytes."

9.0 QUALITY CONTROL

9.1 Follow the manufacturer’s instructions for the quality control procedures specific to use of the testing product. Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

9.2 Energy calibration check -- To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting,

which would indicate drift within the instrument. As discussed in Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 °F).

9.2.1 The energy calibration check should be run at a frequency consistent with manufacturer's recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.2 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank samples -- Two types of blank samples should be analyzed for FPXRF analysis, specifically, instrument blanks and method blanks.

9.3.1 An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window. The instrument blank can be silicon dioxide, a polytetraflurorethylene (PTFE) block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the established lower limit of detection should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lowest level of detection or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration verification checks -- A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision measurements -- The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent. If both in situ and intrusive analytical techniques are used during the course of one day, it is recommended that separate precision calculations be performed for each analysis type.

The equation for calculating RSD is as follows:

$$RSD = (SD/Mean\ Concentration) \times 100$$

where:

RSD = Relative standard deviation for the precision measurement for the analyte
SD = Standard deviation of the concentration for the analyte
Mean concentration = Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the sensitivity, but decreases sample throughput.

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of

replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

9.7 Confirmatory samples -- The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on project-specific data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument calibration -- Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments, namely: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental parameters calibration -- FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are necessary, although site-specific samples with confirmed and validated analytical results for all elements present could be used.

- Cost is reduced because fewer confirmatory laboratory results or calibration standards are necessary.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Sec. 7.3. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective energy FP calibration -- The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference
C_k = Certified concentration of standard sample
C_s = Measured concentration of standard sample

10.2.2 BFP calibration -- BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended

count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical calibration -- An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Sec. 7.3; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is necessary. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are necessary to perform an adequate empirical calibration. The exact number of standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton normalization method -- The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline reading. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later during analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, remove any large or nonrepresentative debris from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Also, the soil surface must be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide example performance data for this method, this modest amount of sample preparation was found to take less than 5 min per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for in situ analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on the desired method sensitivity. Due to the heterogeneous nature of the soil sample, in situ analysis can provide only “screening” type data.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 g or 250 cm^3, which is enough soil to fill an 8-ounce jar. However, the exact dimensions and sample depth should take into consideration the heterogeneous deposition of contaminants and will ultimately depend on the desired project-specific data quality objectives. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Sec. 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the time necessary for homogenization procedure using the fluorescein dye ranged from 3 to 5 min per sample. As demonstrated in Secs. 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, the direct analysis through the plastic bag is possible without the more labor intensive steps of drying, grinding, and sieving given in Secs. 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps should be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 g) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hr in the convection or toaster oven at a temperature not greater than 150 °C. Samples may also be air dried under ambient temperature conditions using a 10- to 20-g portion. Regardless of what drying mechanism is used, the drying process is considered complete when a constant sample weight can be obtained. Care should be taken to avoid sample cross-contamination and these measures can be evaluated by including an appropriate method blank sample along with any sample preparation process.

CAUTION: Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 min per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the established lower limit of detection of the procedure or DQOs of the analysis. If all recommended sample preparation steps are followed, there is a high probability the desired laboratory data quality may be obtained.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in ppm and can be downloaded to a personal computer, which can be used to provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation. See the manufacturer's instructions regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The sections to follow discuss three performance evaluation factors; namely, precision, accuracy, and comparability. The example data presented in Tables 4 through 8 were generated from results obtained from six FPXRF instruments (see Sec. 13.3). The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from "nondetect" to tens of thousands of mg/kg. These data are provided for guidance purposes only.

13.3 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode

detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.4 All example data presented in Tables 4 through 8 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.5 Precision measurements -- The example precision data are presented in Table 4. These data are provided for guidance purposes only. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from "nondetects" to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 4 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the lower limit of detection for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 4. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the lower limit of detection so that an RSD value calculated at 5 to 10 times this limit was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 5 shows these results. These data are provided for guidance purposes only. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the lower limit of detection for the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the in situ homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 5 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square,

measurements of different soil samples were actually taking place within the square. Table 5 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five instead of ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy measurements -- Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 6 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 6 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 6. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 6.

Table 7 provides a more detailed summary of accuracy data for one particular FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. These data are provided for guidance purposes only. Table 7 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability -- Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 8. Similar trends in the data were seen for all instruments. These data are provided for guidance purposes only.

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not

ground; and preparation 4–intrusive, with sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 8 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 8 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Sec. 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time necessary to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 min. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 min per sample. Lastly, when grinding and sieving is conducted, time has to be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 A. D. Hewitt, "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis," American Environmental Laboratory, pp 24-32, 1994.

13.8.2 S. Piorek and J. R. Pasmore, "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer," Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals, Las Vegas, Nevada, February 24-26, 1993, Vol 2, pp 1135-1151, 1993.

13.8.3 S. Shefsky, "Sample Handling Strategies for Accurate Lead-in-soil Measurements in the Field and Laboratory," *International Symposium of Field Screening Methods for Hazardous Waste and Toxic Chemicals*, Las Vegas, NV, January 29-31, 1997.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, http://www.acs.org.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex, X-MET 920 User's Manual.

2. Spectrace Instruments, "Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction," 1994.

3. TN Spectrace, Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.

4. Unpublished SITE data, received from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

Analyte	Chemical Abstract Series Number	Lower Limit of Detection in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: Refs. 1, 2, and 3
These data are provided for guidance purposes only.

TABLE 2

SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	432	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Refs. 1, 2, and 3

TABLE 3

SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Ref. 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4

EXAMPLE PRECISION VALUES

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the Lower Limit of Detection					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84[a]	NR	24.80[a]	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85[a]	NR	24.92[a]	20.92[a]	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69[a]	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32[a]	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

These data are provided for guidance purposes only.
Source: Ref. 4

[a] These values are biased high because the concentration of these analytes in the soil samples was near the lower limit of detection for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the established lower limit detection.

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium[a]	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel[a]	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver[a]	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

These data are provided for guidance purposes only.
Source: Ref. 4
[a] These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.
ND Not detected.
NR Not reported.

TABLE 6

EXAMPLE ACCURACY VALUES

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Ref. 4. These data are provided for guidance purposes only.
n: Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD: Standard deviation; NA: Not applicable; only two data points, therefore, a SD was not calculated.
%Rec.: Percent recovery.
-- No data.

TABLE 7

EXAMPLE ACCURACY FOR TN 9000[a]

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Ref. 4. These data are provided for guidance purposes only.
[a] All concentrations in milligrams per kilogram.
%Rec.: Percent recovery; ND: Not detected; NA: Not applicable.
-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY[1]

	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

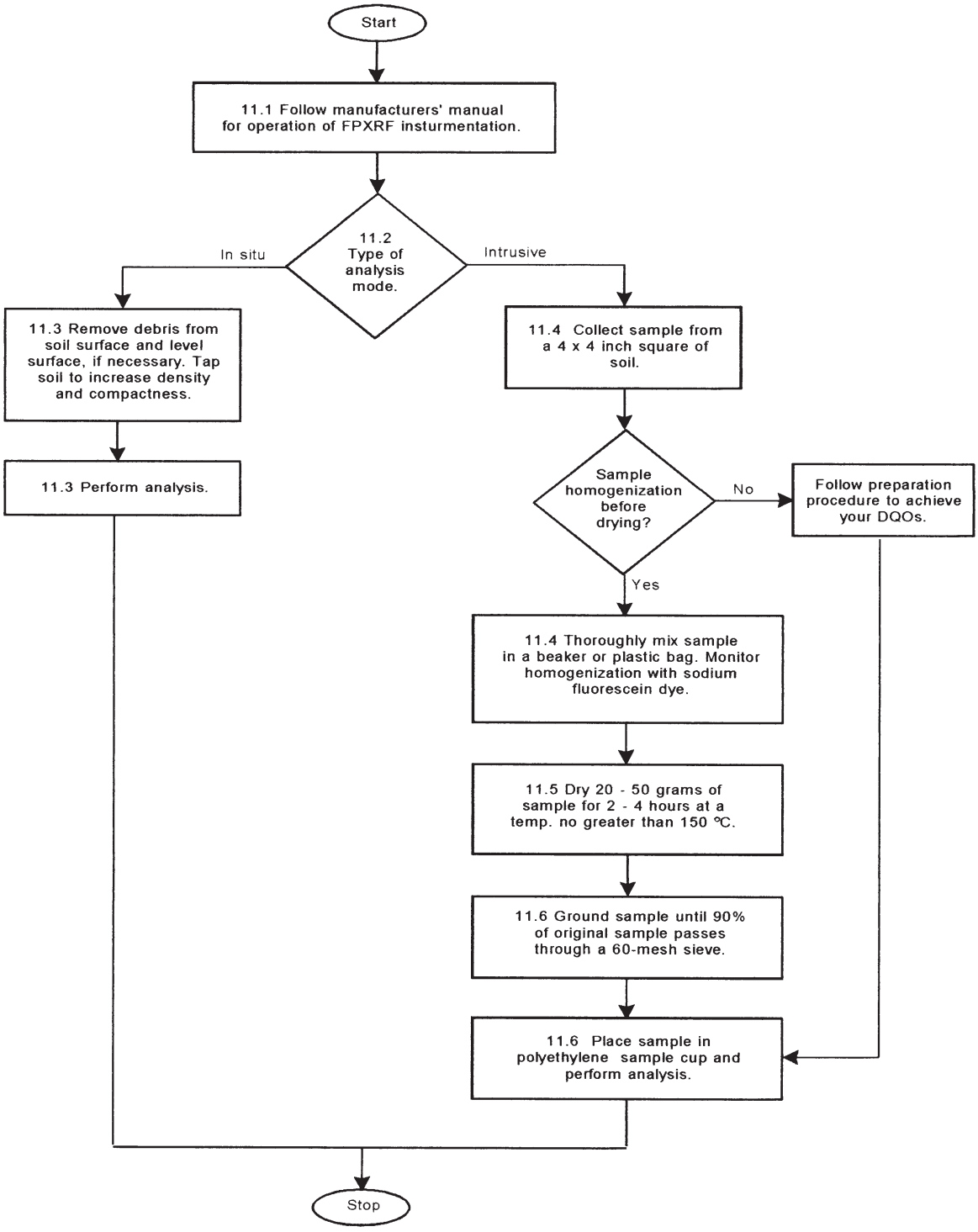
Source: Ref. 4. These data are provided for guidance purposes only.

[1] Log-transformed data

n: Number of data points; r^2: Coefficient of determination; Int.: Y-intercept

— No applicable data

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



STANDARD OPERATING PROCEDURE

No. 2420.6E

SAMPLE CONTAINER SELECTION, PRESERVATION AND HOLDING TIMES

March 14, 2006

By Larry Marchin
ENSV/RLAB/CATS

APPROVED:

Nicole R. Robley — Peer Reviewer — 3/22/06 — Date

[signature] — Laboratory Manager — 23 March 2006 — Date

Harold G. Brown — Independent QA Reviewer — 3/23/06 — Date

Recertified:

Reviewer					
Date					

Table of Contents

Attachments

A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the selection of the proper sample containers and intermediate sample collection containers or devices when collecting samples for specific constituents (parameters) or groups of constituents; and for determining sample preservation and the holding times of samples from the time of collection until analysis is performed.

The guidance contained herein is applicable to all personnel who collect environmental samples for analysis by the Environmental Services Division (ENSV), including EPA and contractor personnel.

B. DEFINITIONS

ASR	Analytical Services Request
BNA	Base-Neutral/Acid Extractable
BOD	Biochemical Oxygen Demand
CBOD	Carbonaceous Biochemical Oxygen Demand
CFR	Code of Federal Regulations
DBCP	1,2-Dibromo-3-chloropropane
DO	Dissolved Oxygen
EDB	Ethylene Dibromide
ENSV	Environmental Services Division
GFF	Glass Fiber Filter
HEM	Hexane Extractable Material
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NPDES	National Pollutant Discharge Elimination System
PCB	Polychlorinated Biphenyl
PE	Performance Evaluation
PM	Project Manager
QC	Quality Control
RLAB	Regional Laboratory Branch
RSTC	Regional Science and Technology Center (facility where the RLAB is located)
RSCC	Regional Sample Control Coordinator
SOP	Standard Operating Procedure
SSR	Sampling Supplies and QC/PE Samples Request
SW	Solid Waste
TCLP	Toxicity Characteristic Leachate Procedure
TKN	Total Kjeldahl Nitrogen

TOX	Total Organic Halogens
TPH	Total Petroleum Hydrocarbons

C. **SAMPLE CONTAINERS**

1. The use of the proper sample container is extremely important to ensure the representativeness of the analytical data obtained, the sufficiency of the sample volume for analysis, and the non-interference or contamination of the sample resulting from the sample container material. When considering the sufficiency of the sample volume for analysis, any matrix spike and matrix spike duplicates (MS/MSD) must be accounted for, and the sampling volume should be adjusted as necessary (One sample for each analyte in each sampling event should at least be double the normal volume or larger volumes for all samples could be used. For example, since water samples for extractables are being collected in 1-gallon bottles, there should be sufficient volume to perform MS/MSD analysis on any sample in the batch.)

2. Special care will be taken to avoid any inadvertent contamination of sample containers prior to or during the sample collection process. Specifically:

   a. Sample containers shall be left sealed or containerized during storage and transport to the sampling location and until the time of actual sample collection. [Exception: Polyethylene cubitainers are received from the manufacturer with the screw caps not attached. The cubitainers are collapsed and nested. The caps are screwed on to the cubitainers after they are filled with sample.]

   b. Sample containers will <u>not</u> be rinsed with the media being sampled during the sample collection process unless specifically required for a given parameter or sampling process (e.g., collecting water samples for toxicity testing).

   Note: The specific SOPs on sample collection should be referred to for the appropriate collection procedure to be used.

3. The sample container selection process is governed by:

   a. The parameter or group of parameters to be analyzed; this includes the desired level of detection in many cases.

   b. The media or matrix to be sampled (i.e., air, solid, tissue, water or other).

c. The analytical method to be used for the analysis.

d. The laboratory (i.e., EPA Region 7 laboratory or a contract laboratory) to perform the analysis. The laboratory where samples will be analyzed is determined by the Laboratory Branch (RLAB) upon receipt of an Analytical Services Request (ASR) form.

4. Attachment 1 provides specific guidance for use in Region 7 in selecting the proper sample container by parameter, or parameter group, and the media being sampled. Additional guidance or requirements for acceptable materials of sample containers are contained, by the parameter to be analyzed, in the current 40 CFR Parts 136 and 141, and SW-846. Also, guidelines are normally found in the specific analytical methods and sampling procedures.

5. When the use of intermediate sample collection containers is necessary, guidance on recommended intermediate container materials may be found in Attachment 2.

D. SAMPLE PRESERVATION

1. The immediate on-site analysis of samples at the time of collection is, in most cases, neither possible nor practical. Therefore, methods have been established to maintain the integrity of the sample until analysis can be accomplished. Even when samples are preserved in an appropriate manner, they should be analyzed as soon as possible after collection. An integral part of preservation is the selection of the proper sample container, the pretreatment of a sample container (if necessary), and the holding time allowable prior to analysis.

2. The purpose of sample preservation is to 1) retard biological action; 2) retard hydrolysis of chemical compounds and complexes; 3) reduce volatility of constituents; and 4) reduce absorption effects. The preservation methods used are generally limited to pH control, chemical addition, refrigeration or freezing. As a rule, the refrigeration (or icing) of samples should be utilized to maintain the samples at a temperature of $4 \pm 2°C$ during sample collection (including the collection of time or flow-weighted composite samples), transport, and storage.

3. The current guidance for sample preservation for use in Region 7 is provided in Attachment 1. Although taken into consideration when preparing this guidance, additional specific guidelines and requirements for sample preservation may be found in regulations (e.g., 40 CFR Part 136), publications (e.g., SW-846) and applicable analytical methods.

4. The following guidance is provided for field personnel to use when preserving the types of samples indicated:

   a. Grab Samples: The applicable preservation method must be accomplished immediately upon sample collection.

   b. Manually Composited Water Samples: The applicable preservation must be added, in full, to the initial aliquot and thus be available for each subsequent aliquot.

   c. Automatically Composited Water Samples:

      (1) When collected for either a single parameter or a parameter group where the type and amount of preservative required are identical, the applicable preservative is added to each container receiving an aliquot, prior to compositing.

      (2) When collecting a composite sample that will later be split to create samples for a variety of individual parameters and each of these parameters requires different preservation methods, the samples collected for the composite should be iced to maintain a temperature of 4°C until the compositing and splitting can be completed. The appropriate preservative is then added at the time the composite is split into separate containers.

5. Samples of the following media will <u>not</u> be preserved with the addition of any chemical compound, but will be chilled to 4°C after collection and during transport and storage.

   a. Solids: soil, sediment, sludge

   b. Tissue (or freeze, -15 to -20°C)

   c. Other: non-aqueous solutions, product samples (liquid or solid), drum samples, wipe samples

6. The following parameters require special procedures:

   a. **Biochemical Oxygen Demand (BOD)/Carbonaceous Biochemical Oxygen Demand (CBOD)**. Water samples of chlorinated effluents collected for analysis of this parameter must be labeled with the word "CHLORINATED" on the sample tag to alert laboratory personnel.

Chlorinated samples require different analytical procedures than unchlorinated samples for this parameter.

b. **Cyanide, Total and Amenable to Chlorination.** Water samples for these parameters should not be collected using automatic samplers, but should be collected manually either as a grab or a composite of several grab samples which are preserved at the time of collection. Since oxidizing agents such as chlorine decompose many cyanides, the sample must be treated to eliminate such agents, if they are present, at the time of collection. The presence of chlorine is determined by testing a drop of the sample with potassium iodide (KI)-starch test paper. A change in the color of the paper to blue indicates the need for treating the sample with a dechlorinization agent. This treatment is accomplished by adding ascorbic acid, a few crystals at a time followed by the subsequent testing of a drop of sample until no color is produced on the KI indicator paper. An additional 0.6 gram of ascorbic acid is then added for each liter of sample volume. Preservation of the sample is then accomplished by adding 2 mL of 10 N sodium hydroxide solution or 10 pellets of sodium hydroxide crystals per liter of sample (to pH $\geq$ 12) and by icing the sample to 4°C during transport and storage.

c. **Dissolved Oxygen (DO).** Water samples for this parameter are collected only on a grab basis. When collecting a sample for this parameter, the sample bottle should be filled to overflowing to ensure that no air bubbles are entrapped in the bottle when the stopper is replaced. When immediate measurement is not possible on site utilizing the DO probe method, the sample will be "fixed" immediately upon collection by first adding 2 ml of manganous sulfate ($MnSO_4$) solution and then 2 ml of alkali-iodide-azide solution well below the surface of the liquid. The sample is mixed by inverting the bottle several times while holding the stopper in place and allowing to set until the floc has settled half way. Carefully remove the stopper and immediately add 2 ml of concentrated sulfuric acid (H_2SO_4) by allowing the acid to run down the neck of the bottle. Re-stopper and mix again and store at 10-20°C out of direct sunlight. Completion of the analysis for DO utilizing the Winkler titration method (Azide Modification) should be accomplished as soon as possible after collection and fixing, but not more than 8 hours after collection.

d. **Metals, Dissolved**. Water samples for this parameter must be filtered on site utilizing a 0.45μm membrane filter as soon as practical after collection and then acidified with 5 mL 1:1 HNO_3 per liter of sample.

e. **Microbiology (Total and Fecal Coliform; Fecal Streptococci).** Water samples for these parameters will be collected only on a grab basis. For chlorinated effluents, sodium thiosulfate (0.008% $Na_2S_2O_3$) is added to the sample. This dechlorinating agent is normally added during sample container preparation and is, therefore, normally present in the sample container. Care must be taken during sample collection to avoid overfilling or rinsing out the agent. In addition, care must be taken to avoid contamination of the sterile sample container prior to or during sample collection; i.e., leave cap on container until ready to collect sample and do not place fingers in container or on the inside of the cap, while collecting a sample. An air space should be left at the top of the container after the sample is collected. Ice to 4°C.

f. **Oil and Grease (Hexane Extractable Material, HEM)** Water samples for this parameter will be collected in one liter glass bottles on a grab basis only and acidified with 1:1 HCl to pH < 2 immediately after collection. The sample container should never be rinsed with the water or wastewater because these constituents tend to adhere to the sides of the container. Care should be taken to avoid contamination of the sample from fingers placed in the container or on the inside liner of the cap. In addition, enough air space should be allowed in the container to allow for the addition of the preservative.

g. **Organics, Volatiles.** Grab samples only are collected for these parameters. Each sample will consist of two (2) 40-mL vials. Generally, four (4) 40-mL vials per water sample will be collected for low detection level and drinking water samples.

(1) Drinking water samples containing residual chlorine must be treated with sodium thiosulfate or ascorbic acid (depending on the analytical method) at the time of collection.

(2) Wastewater samples containing residual chlorine must be treated with ascorbic acid (25 mg per 40 mL) at the time of collection. These dechlorinating agents must be placed in the vials prior to collecting the samples.

(3) When collecting water samples, fill the sample bottles to overflowing, but take care not to flush out the sodium thiosulfate or ascorbic acid, if present. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. After collection, the pH of the sample is adjusted

to a pH < 2 by carefully adding one drop of 1:1 HCl for each 20 mL of sample volume. Seal the sample bottles teflon-face down, and shake vigorously for 1 minute. A proper seal can be checked by inverting the sample and lightly tapping the end on a solid surface. If air bubbles are present, open the vial, add additional sample, reseal, and recheck for air bubbles. Store samples out of direct sunlight.

h. **Phenols (Phenolics)**. Water samples for this parameter should not be collected by using automatic samplers, but should be collected as a grab or a manual composite of grabs and preserved with 2 mL H_2SO_4 to a pH < 2 at the time of collection. The samples should be iced to maintain them at 4°C during transport and storage.

E. SAMPLE HOLDING TIMES

1. The issue of holding times for samples is critical in the sample collection and analysis process, because the integrity of the samples can be affected depending on the parameter to be analyzed. Sample holding times are defined as the period of time between sample collection and initiation of sample analysis. In the case of timed composite samples, the holding time starts at the end of the compositing period (i.e., at the time the last portion of the composite sample is obtained).

2. Since holding times can affect the validity of the reported analytical results (especially in certain media programs and in enforcement actions), everyone involved in planning and executing sampling activities; planning and performing analyses; and reviewing analytical results must be cognizant of the implications of exceeding them during the process. In many instances, the holding times are required by specific regulations (e.g., 40 CFR Part 136 for wastewater samples under the NPDES program), while many others are recommended. Also, see Footnote 8 of Attachment 1.

3. Although many of the holding times contained in Attachment 1 were derived from regulatory requirements, the holding times should be considered as guidelines. When making decisions on the validity of analytical results based on holding times, personnel should consult appropriate regulations to determine if there are specific requirements for sample holding times.

F. OBTAINING SAMPLING SUPPLIES

1. When sampling supplies (e.g., sample containers, sample collection devices, preservatives, etc.) are needed, the project manager for the specific sampling

activity requests the necessary supplies on the Sampling Supplies and QC/PE Samples Request (SSR).

2. The requestor can pick up the supplies at the ENSV warehouse facility (3150 Dodge). The preservatives must be picked up at the RSTC (300 Minnesota Avenue). It is recommended that the requestor contact the Regional Sample Control Coordinator (RSCC) or designated back-up at the RLAB before going to either facility to ensure the supplies are ready for issuance.

G. REFERENCES

1. Code of Federal Regulations, Title 40 (40 CFR), Part 136.

2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. EPA.

3. Standard Methods for the Examination of Water and Wastewater, Joint Editorial Board, American Public Health Association, American Water Works Association and Water Environment Federation, Latest Edition.

Attachment 1

Guide for Sample Container Selection, Preservation and Holding Times

Parameter[1]	Sample Container[2]	Preservation	Holding Time[8]
I. **Air and Gaseous**			
Dioxins/Furans	Puff glass jar	Cool 4°C	365 days
Metals on High-Vol GFF	1-gal. resealable plastic bag	Ambient Temperature	
Ozone Precursors			
Ambient levels	6-L canister	None	60 days
Source levels	400 mL canister	None	60 days
Particulate Matter on High-Vol GFF	1-gal. resealable plastic bag	Ambient Temperature	
Pesticides/PCBs	Puff glass jar	Cool 4°C	7 days
Semivolatiles/BNA	Puff/XAD glass jar	Cool 4°C	14 days
Volatile Organics			
Ambient levels	6-L canister	None	60 days
Source levels	400 mL canister	None	60 days
GC/MS Scan	6-L canister	None	30 days
II. **Soil, Sediment and Solids**			
A. Non-Product Samples			
Asbestos	Plastic jar	None	None
Cyanide	8-oz. glass jar	Cool 4°C	28 days
Dioxin/Furans	8-oz. glass jar/sealable plastic bag in 1-qt. paint can	Cool 4°C	365 days
Explosives	8-oz. glass jar	Cool 4°C	14 days
Flashpoint	8-oz. glass jar	Cool 4°C	
GC/MS Scan, SemiVOA	8-oz. glass jar	Cool 4°C, store in dark	14 days
GC/MS Scan, VOA	2, 40mL VOA vials	Cool 4°C	14 days
Herbicides	8-oz. glass jar	Cool 4°C	14 days
Metals			
All metals except Cr^{+6} and Hg, collected separately	8-oz. glass jar	Cool 4°C	180 days
Mercury	8-oz. glass jar	Cool 4°C	180 days
Cr^{+6}	8-oz. glass jar	Cool 4°C	30 days
Methanol	8-oz. glass jar	Cool 4°C	14 days
Nutrients			
Nitrogen (NH_3, NO_2, TKN)	8-oz. glass jar	Cool 4°C	28 days
Phosphorous, total	8-oz. glass jar	Cool 4°C	
Oil and Grease	8-oz. glass jar	Cool 4°C	
pH	8-oz. glass jar	Cool 4°C	None
Phenolics (colorimetric)	8-oz. glass jar	Cool 4°C	28 days
Perchlorate	8-oz. glass jar	Cool 4°C	28 days
Pesticides/PCBs	8-oz. glass jar	Cool 4°C	14 days
Radioactivity	8-oz. glass jar	Cool 4°C	180 days

Parameter	Sample Container	Preservative	Holding Time
II. Soil, Sediment, and Solids (cont.)			
A. Non-Product Samples (cont.)			
Semivolatiles/BNA	8-oz. glass jar	Cool 4°C	14 days
Sulfate/Sulfide	8-oz. glass jar	Cool 4°C	28 days
Soil Toxicity Test	1-gal. ziplock bag	Cool 4°C	56 days
Total Organic Carbon	8-oz. glass jar	Cool 4°C	28 days
Total Petroleum Hydrocarbons (TPH)			
SemiVOA	8-oz. glass jar	Cool 4°C	14 days
VOA	2, 40-mL glass vials	Cool 4°C	14 days
Total Kjeldahl Nitrogen	8-oz. glass jar	Cool 4°C	None
Organic Parameters			
All except volatile organics	8-oz. glass jar	Cool 4°C	14 days
Volatile organics[3]	2, 40-mL glass vials	Cool 4°C or MeOH & Cool 4°C or $NaHSO_4$ & Cool 4°C	14 days
TCLP			
Metals, except Hg	8-oz. glass jar	Cool 4°C	180 days to extract, 180 days after extraction (required)
Mercury	8-oz. glass jar	Cool 4°C	28 days to extract, 28 days after extraction (required)
Volatile organics	2, 40-mL glass vials	Cool 4°C	14 days to extract, 14 days after extraction (required)
Semivolatile organics	8-oz. glass jar	Cool 4°C	14 days to extract, 7 days to extraction, 40 days after extraction (required)
Pesticides/Herbicides	8-oz. glass jar	Cool 4°C	14 days
B. Product Samples			
All parameters	8-oz. glass jar/sealable plastic bag in paint can	Cool 4°C	
III. Tissue			
Fish, collected for whole body/edible portion, all parameters	Double wrapped in heavy duty foil	Freeze	
Resectioned tissue, collected for:			
Metals	Double wrapped in heavy duty foil	Freeze	180 days
Semivolatiles	Double wrapped in heavy duty foil	Freeze	
Volatiles	Double wrapped in heavy duty foil	Freeze	
Foliage			
Herbicides/Pesticides	Double wrapped in heavy duty aluminum foil	Freeze	
Macroinvertibrates[6]		70% ethanol	6 months
Periphyton[6]			
Chlorophyll A		Freeze, store in dark	30 days
Enumeration		5% formalin, Cool 4°C, store in dark	6 months
Dioxins/Furans	Double wrapped in heavy duty foil	Freeze	365 days
Phytoplankton, collected for[6]			
Chlorophyll A		Cool 4°C, store in dark	14 days
Enumeration		5% formalin	6 months

Parameter	Sample Container	Preservative	Holding Time
IV. Aqueous Samples			
Chlorine Dioxide	1-L plastic cubitainer	Cool 4°C	None
Chlorophyll A	4-L plastic cubitainer	Cool 4°C, store in dark	14 days
Coliform, fecal	300-mL sterile plastic bottle	Cool 4°C, 0.008% $Na_2S_2O_3$	6 hours
Dioxins/Furans	1-L amber glass bottle	Cool 4°C	365 days
Dissolved Organic Carbon	1-L amber glass bottle	H_2SO_4 to pH<2 Cool 4°C	28 days
Explosives	128-oz. amber glass bottle	Cool 4°C	7 days to extract, 40 days after extraction
Herbicides	128-oz. amber glass bottle	Cool 4°C	7 days to extract, 40 days after extraction
Flashpoint	8-oz. glass jar	Cool 4°C	
Metals (except Hg and Cr^{+6})			
Total and acid soluble	1-L plastic cubitainer	HNO_3 to pH<2	6 months
Dissolved	1-L plastic cubitainer	Filter HNO_3 to pH<2	6 months
Chromium, hexavalent	1-L plastic cubitainer	Cool 4°C	24 hours
Mercury	1-L plastic cubitainer	HNO_3 to pH<2	28 days
Strontium	1-L plastic cubitainer	HNO_3 to pH<2	6 months
Acid, %	1-L plastic cubitainer	Cool 4°C	None
Alkalinity	1-L plastic cubitainer	Cool 4°C	14 days
BOD/CBOD	1-L plastic cubitainer	Cool 4°C	48 hours
COD	1-L plastic cubitainer	H_2SO_4 to pH<2 Cool 4°C	28 days
Chlorine (residual)	1-L plastic cubitainer	Cool 4°C	1 day
Conductivity	1-L plastic cubitainer	Cool 4°C	28 days
Cyanide (total and amenable To chlorine)	1-L plastic cubitainer	(Ascorbic acid), NaOH to pH>12, Cool 4°C	14 days
Halides (Br^-, Cl^-, F^-)	1-L plastic cubitainer	Cool 4°C	28 days
Haloacetic Acids/Dalapon	1-L amber glass bottle	Ammonium Chloride, Cool 4°C	14 days
Hardness	1-L plastic cubitainer	HNO_3 to pH<2, Cool 4°C	6 months
Inorganic Anions	1-L plastic cubitainer	EDA, Cool 4°C	14 days
Methane, Ethane, Ethene	2, 40-mL VOA vials	Cool 4°C	7 days
Methanol	1-L amber glass bottle	Cool 4°C	7 days
Nonfilterable Solids (NFS)	1-L plastic cubitainer	Cool 4°C	7 days
Oxygen, Dissolved (Winkler)	300-mL glass BOD bottle with Attached ground glass stopper	$MnSO_4$ + Alkali-Iodide-Azide, H_2SO_4	8 hours
Dissolved Oxygen, probe Method	1-L plastic cubitainer	Cool 4°C	1 day

Parameter	Sample Container	Preservative	Holding Time
IV. Aqueous Samples (cont.)			
pH	1-L plastic cubitainer	Cool 4°C	determine immediately
Perchlorate	1-L plastic cubitainer	Cool 4°C	28 days
Residue			
All but settleable	1-L plastic cubitainer	Cool 4°C	7 days
Settleable	1-L plastic cubitainer	Cool 4°C	48 hours
Sulfate	1-L plastic cubitainer	Cool 4°C	28 days
Sulfide	1-L plastic cubitainer	Zinc acetate + NaOH to pH>9, Cool 4°C	7 days
Total Dissolved Solids (TDS)	1-L plastic cubitainer	Cool 4°C	7 days
Total Kjeldahl Nitrogen	1-L plastic cubitainer	H_2SO_4 to pH<2.5, Cool 4°C	28 days
Total Solids	1-L plastic cubitainer	Cool 4°C	7 days
Turbidity	1-L plastic cubitainer	Cool 4°C	48 hours
Nutrients			
Nitrogen-Ammonia	1-L plastic cubitainer	H_2SO_4 to pH<2 , Cool 4°C	28 days
Nitrogen-Organic	1-L plastic cubitainer	H_2SO_4 to pH<2, Cool 4°C	28 days
Nitrate	1-L plastic cubitainer	Cool 4°C	48 hours
Nitrate-Nitrite	1-L plastic cubitainer	H_2SO_4 to pH<2, Cool 4°C	28 days
Nitrite	1-L plastic cubitainer	Cool 4°C	48 hours
Phosphorous (total)	1-L plastic cubitainer	H_2SO_4 to pH<2.5, Cool 4°C	28 days
Ortho-phosphate	4-oz. plastic bottle	Filter, Cool 4°C	48 hours
Phosphorous, (dissolved)	1-L plastic cubitainer	Filter, H_2SO_4 to pH<2, Cool 4°C	28 days
Carbamates[10]	60-mL screw cap vial	1.8 mL monochloroacetic acid buffer pH<3, Cool 4°C	14 days
Oil and Grease (HEM)	1-L glass jar	1:1 HCL to pH<2, Cool 4°C	28 days
Pesticides/PCBs	128-oz. amber glass bottle	Cool 4°C	7 days to extract, 40 days after extraction[9]
Phenolics	1-L glass jar	H_2SO_4 to pH<2, Cool 4°C	28 days
Radionuclides	1-L plastic cubitainer	HNO_3 to pH<2	6 months (water:alpha/beta) 5 days(gamma; DW:alpha/beta)
Semivolatile/BNA	128-oz. amber glass bottle	Cool 4°C, store in dark	7 days to extract, 40 days after extraction
GC/MS Scan (BNA)	128-oz. amber glass bottle	Cool 4°C, store in dark	7 days
Total Organic Carbon (TOC)	1-L amber glass bottle	H_2SO_4 to pH<2, Cool 4°C	28 days
Total Organic Halogens (TOX)	8-oz. amber glass bottle	Cool 4°C	7 days

Parameter	Sample Container	Preservative	Holding Time
IV. Aqueous Samples (cont.)			
Total Petroleum Hydrocarbons (TPH)			
SemiVOA	128-oz. amber glass bottle	Cool 4°C	7 days
VOA	2, 40-mL VOA vials	Cool 4°C	14 days
Toxicity Tests			
Acute	10-L (2.5-gal) plastic cubitainer	Cool 4°C	36 hours
Bioscreen (24 or 48 hr.)	4-L (1-gal.) plastic cubitainer or glass bottle	Cool 4°C	24 hours
Chronic	20-L (2.5/5-gal.) plastic cubitainer	Cool 4°C	36 hours
Triazine Herbicides	128-oz. amber glass bottle	Cool 4°C	14 days
Tritium	8-oz. glass jar	None	5 days
Volatile Organics[3, 5]			
Purgeable halocarbons	2, 40-mL glass vials	HCl to pH<2, Cool 4°C	14 days
Purgeable aromatic hydrocarbons	128-oz. amber glass bottle	HCl to pH<2, Cool 4°C	7 days
Routine Detection Level	2, 40-mL glass vials	HCl to pH<2, Cool 4°C	14 days
Low Detection Level	4, 40-mL glass vials	HCl to pH<2, Cool 4°C	14 days
EDB/DBCP	2, 40-mL glass vials	Sodium Thiosulfate Cool 4°C	14 days
GC/MS Scan (VOA)	2, 40-mL VOA vials	HCl to pH<2, Cool 4°C	14 days
V. Liquid, Non-Aqueous			
TCLP (> 5% solids)[4]			
Mercury	8-oz. glass jar	Cool 4°C	28 days to TCLP extract, 28 days after extraction
Metals, except Hg	8-oz. glass bottle	Cool 4°C	180 days
Volatile Organics	2, 40-mL glass vials	Cool 4°C	14 days to TCLP extract, 14 days after extraction
Semivolatile Organics	8-oz. glass jar	Cool 4°C	14 days to TCLP extract, 7 days to extraction, 40 days after extraction
Pesticides/Herbicides	128-oz. amber glass bottle	Cool 4°C	7 days
Organic Parameters			
All except volatile organics	8-oz glass jar	Cool 4°C	7 days to extract, 40 days after extraction
Volatile organics	8-oz glass jar	Cool 4°C	14 days
VI. Wipe Samples[7]			
Arsenic	8-oz. glass jar	Cool 4°C	
Cyanide	8-oz. glass jar	Cool 4°C	
Dioxin	8-oz. glass jar	Cool 4°C	365 days
Herbicides/Pesticides	8-oz. glass jar	Cool 4°C	14 days
Metals	8-oz. glass jar	Cool 4°C	180 days
Picric Acid	8-oz. glass jar	Cool 4°C	
Organic other parameters			
All except volatile organics	8-oz. glass jar	Cool 4°C	7 days to extract, 40 days after extraction
Volatile organics	8-oz. glass jar	Cool 4°C	

Parameter	Sample Container	Preservative	Holding Time
VI. Wipe Samples (cont.)			
TCLP (> 0.5% solids)			
Metals, except mercury	8-oz. glass jar	Cool 4°C	180 days to TCLP extract, 180 days after extraction

[1] Non-product and product sample definitions: A non-product sample is a sample which consists primarily of naturally occurring materials that may contain mechanically or chemically manufactured materials or substances as contaminants. A product sample is a sample which is known to consist primarily of a mechanically or chemically manufactured material that does not otherwise occur naturally in the immediate environment being sampled. A sample which cannot be identified as a non-product sample should be considered a product sample

[2] All glass containers require a Teflon-lined lid or cap.

[3] These parameters are always collected as replicates. The sample container consists of two or four 40-mL glass vials (VOA vials) and an activated carbon filled thimble contained in a 1-L plastic cubitainer.

[4] § 40 CFR Part 261 Appendix II, 2.1: For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

[5] Sodium Thiosulfate ($Na_2S_2O_3$) or Ascorbic Acid ($C_6H_8O_6$) is utilized to de-chlorinate samples of chlorinated water or wastewater prior to pH adjustment. For drinking water samples, consult the applicable method to determine appropriate dechlorinating agent.

[6] Sample containers, preservation, and holding times vary. The information provided is Regional guidance. For compliance samples, consult appropriate references for complete preservation requirements.

[7] Each jar should contain a medical gauze pad.

[8] Generally, there are no required holding limits for air, soil, product and wipe media. (Exceptions: see Chapter 4 of SW-846). However, it is recommended that samples be analyzed within the holding time limits established in aqueous media for the specific analytes or analyte groups.

[9] Method 505 holding time 14 days (7 days for Heptachlor), preserve with Sodium Thiosulfate, 4° C, container 40-mL vial.

[10] Method 531.1 samples must be preserved to a pH<3 using monochloroacetic acid to minimize degradation of oxamyl, 3-hydroxycarbofuran, aldicarb sulfoxide, and carbaryl in neutral and basic waters. If residual chlorine is present add 80 mg of sodium thiosulfate per liter of sample to the sample bottle prior to collecting the sample.

Attachment 2

Guide for Selecting Intermediate Sample Container Material

Media Sampled/Parameter	Intermediate Sample Container Material
I. Soil, Sediment and Solids	
A. Non-Product and Product Samples	
All parameters except volatile organics	Glass, Aluminum
Volatile Organics[1]	Glass, Stainless Steel, Aluminum
II. Tissue	
A. Fish	
All parameters	Glass, Plastic
III. Liquids	
A. Aqueous Samples	
Chlorophyll A	Artificial substrate (glass slide)
Coliform, fecal[2]	Glass or Plastic
Dioxin/Furans	Glass, Stainless Steel (solvent rinsed)
Explosives	Glass
Metals:	
All except Cr^{+6}	Glass, Plastic, Automatic sampler equipped with Tygon intake tubing and glass or plastic compositing container.
Chromium, hexavalent	Glass, Plastic
Minerals and Dissolved Materials:	
Acid (%), Alkalinity, BOD, Chloride, Conductivity, Hardness, Residue, Sulfate, Turbidity	Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Chlorine	Glass, Plastic, Stainless steel
COD	Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Cyanide	Glass, Plastic, Stainless steel

Media Sampled/Parameter	Intermediate Sample Container Material
III. Liquids (cont.)	
A. Aqueous Samples (cont.)	
Fluoride	Plastic, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and plastic compositing container
Oxygen, dissolved	Glass, Plastic, Stainless steel
pH, lab or field	Glass, Plastic, Stainless steel, Teflon
Sulfide	Glass, Plastic, Stainless steel
Nutrients (N & P)	Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Oil and Grease[2]	Glass
Pesticides/PCBs	Glass, Stainless steel, Automatic sampler equipped with Teflon intake tubing and glass compositing container (cleaned and solvent rinsed)
Phenols/Phenolics	Glass, Plastic, Stainless steel
Radionuclides	Glass, Plastic
Semivolatiles/BNA	Glass, Stainless steel, Automatic sampler equipped with Teflon intake tubing and glass compositing container (cleaned and solvent rinsed)
Total Organic Carbon	Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Total Organic Halogens	Glass, Plastic
Toxicity Tests: Acute Bioscreen Chronic	Automatic sampler equipped with Teflon or new Tygon tubing, and glass or Nalgene compositing container (see SOP No. 2334.6 for tubing and container cleaning)
Volatile Organics[1]	Glass, Stainless steel (not solvent rinsed)
B. Non-Aqueous Product Samples	
All parameters	Glass

[1]An intermediate sample collection device is not recommended for this parameter, but, if one is necessary, care must be taken to insure that the device has not been solvent rinsed.

[2]An intermediate container is not recommended for this parameter; therefore, every effort must be made to collect the sample directly into the sample container.

STANDARD OPERATING PROCEDURE

No. 2420.1E

SAMPLE RECEIPT AND LOG-IN

March 31, 2006

by

Nicole Roblez
ENSV/RLAB/CATS

APPROVED:

Peer Reviewer — Date: 3-30-06

Laboratory Manager — Date: 30 March 2006

Independent QA Reviewer — Date: 3/31/06

Recertified:

Reviewer					
Date					

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ATTACHMENTS:

A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to establish a uniform policy and procedure for the receipt and log-in of environmental samples shipped by Environmental Protection Agency (EPA) field personnel or their contractors to the Environmental Services Division (ENSV) Regional Laboratory (RLAB). The policies and procedures contained in this SOP are applicable to all ENSV personnel and EPA contractors.

B. SUMMARY OF METHOD

Sample shipments, normally in ice chests (coolers), are delivered on a daily basis by either field personnel or air courier. In some instances, samples are shipped via over night carrier (e.g. Federal Express (FedEx) or Airborne Express) to the ENSV RLAB.

The Regional Sample Control Coordinator (RSCC) (or designated backup) receives all samples. The RSCC's sample custodial duties reside in the Contracts and Technical Support (CATS) Section, RLAB/ENSV. The RSCC (or designated backup) is available to receive samples Monday through Friday (excluding holidays) from 7:30 a.m. until 4 p.m. Deliveries on days and times other than these require scheduling through the RLAB Program Manager. However, RLAB does maintain a secured refrigerator in the receiving area for night and weekend deliveries allowing the Project Manager to temporarily store samples until they can relinquish them to the RSCC (or designated backup). When samples are left in that refrigerator, it is the Project Manager's responsibility to notify the RSCC (or designated backup) that samples are stored there (e.g., voice mail message or written note).

The RSCC (or designated backup) coordinates delivery of sample shipments with field personnel. When samples arrive, the sample collector, Purchasing and Receiving, or other ENSV personnel will contact the RSCC (or designated backup) who will then proceed to the sample receiving area.

During this time, either the Purchasing and Receiving person (or designated backup) or RSCC (or designated backup) will perform the radioactive materials cooler/sample survey according to SOP 2420.15, Management of Radioactive Materials in the Laboratory Environment.

Personnel delivering samples to the RLAB are to observe the radioactive materials survey and sample receipt procedures for the purpose of alleviating any questions and/or problems relating to the radioactive materials survey, samples or paperwork.

No samples are to be "dropped-off" without the appropriate notification to the RSCC (or designated backup). Any such samples will not be processed until the Project Manager contacts the RSCC (or designated backup).

All samples delivered must have an associated Analytical Services Request (ASR) form on file (Attachment 1 contains a copy of the ASR form that is currently in use). The only exception to this will be for emergency spills or special enforcement actions that cannot legitimately be pre-planned. Acceptance of such samples will be on a case-by-case basis with specific approval of the RLAB Program Manager. For samples of this nature, the Project Manager must initiate and be in the process of routing the ASR for signature/acceptance concurrent to sample delivery.

C. DEFINITIONS\ACRONYMS

ANSETS	Analytical Services Tracking System
ASR	Analytical Services Request
BODs	Biological Oxygen Demand
CATS	Contracts and Technical Support
CLP	Contract Laboratory Program
COC	Chain Of Custody Record
ENSV	Environmental Services Division
EPA	Environmental Protection Agency
EPA TO#	REAP EPA Task Order Number
F2L	CLP Forms2Lite5.1
FedEx	Federal Express
R7LIMS	Laboratory Information Management System
PM	Project Manager
PO	Project Officer
Project	Sampling effort
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
REAP	Region 7 Environmental Analysis Program
ESAT	Region 7 Environmental Services Assistance Team
RLAB	Regional Laboratory

RQAM	Regional Quality Assurance Manager
RSCC	Regional Sample Control Coordinator
RSTC	Region 7 Science and Technology Center
SMO	Sample Management Office
SOP	Standard Operating Procedure
SRN	Sample Receipt Notification
Tags	Sample container labels
TAT	Turn-around Time
VOAs	Volatiles

D. PERSONNEL QUALIFICATIONS

Personnel performing this task should have a basic knowledge of RLAB sample management procedures and the computer software utilized.

1. Responsibilities of Personnel Performing this Task:

   a. Project Manager

      (1) The Project Manager submits a completed ASR to the RLAB at least 30 days before the projected sampling delivery date.

      (2) The Project Manager collects and ships properly labeled, preserved, and packaged samples to ENSV in a timely fashion.

      (3) The Project Manager is responsible for the accuracy and completeness of all accompanying paperwork. If any changes are required as a result of the sampling (e.g., sample number changes, additional analytes, samples not collected, quality control (QC) code additions, etc.), the Project Manager must see that these corrections are made on all paperwork. All changes made to the paperwork (sample tags, field sheets) must also be made to the information contained in the Laboratory Information Management System (LIMS). It is the responsibility of the Project Manager to supply correct information so that the RSCC (or designated backup) can reconcile the samples in LIMS. Whenever possible, any changes are made prior to the delivery of the samples. The Project Manager coordinates the changes through the appropriate person as indicated. If necessary, the RSCC (or designated backup) will assist the Project Manager when changes are noted prior to

sample collection/delivery, concurrent to sample delivery or after. The Project Manager is available to help resolve any problems with his samples or designates someone to do this for him in his absence. This requires that when delivering samples, the Project Manager stays with the RSCC (or designated backup) to answer any questions. Samples are not to be dropped off without notification to the RSCC (or designated backup) or sample receiving personnel. The Project Manager calls the RSCC (or designated backup) close to the anticipated delivery time of samples sent in by air courier (e.g., FedEx) to confirm that samples have arrived and to answer any questions the RSCC (or designated backup) may have. In the case that the Project Manager is not available and has no alternate, RLAB contacts the Project Manager's supervisor for any necessary reconciliation information in order to resolve problems quickly and initiate sample processing.

b. Regional Sample Control Coordinator

(1) The RSCC (or designated backup) performs the radioactive materials cooler/sample survey according to SOP 2420.15, Management of Radioactive Materials in the Laboratory Environment if the Purchasing and Receiving person (or designated backup) has not performed this task.

(2) The RSCC (or designated backup) verifies the presence of all samples, checks all documentation, and signs the Chain Of Custody Record (COC) after all paperwork is complete and accurate.

(3) The RSCC (or designated backup) works with the Project Manager to obtain correct information and put the amended information into LIMS.

(4) The RSCC (or designated backup) notifies the Project Manager of problems which prevent acceptance of the samples by ENSV. RLAB maintains all samples received in a secure location including those pending reconciliation of problems.

(5) The RSCC (or designated backup) is responsible for the proper storage, tracking and/or distribution of the samples to the appropriate in-house and contract laboratories (this includes while the sample is in transit to the contract laboratory facility). Refer to SOP 2420.2 for sample storage procedures.

(6) The RSCC (or designated backup) logs samples into LIMS and prepares an electronic sample receipt notification to inform RLAB analytical personnel that the samples have been delivered to the RSTC and the location of the refrigerators in which they have been placed.

E. PROCEDURAL STEPS

1. Sample Receipt Procedure

a. The RSCC (or designated backup) is notified of sample receipt through periodic checks of the sample receiving area and through notification by sampling or ENSV personnel. The RSCC (or designated backup) identifies the project and ASR number from the COC and field sheets, and locates a copy of the ASR.

(1) If all paperwork is sealed with the samples in the cooler (often taped to the inside lid), the RSCC (or designated backup) must open the cooler(s) (see Section E.4.), in order to identify the ASR number.

(2) Except under extraordinary circumstances, the RSCC (or designated backup) must have a copy of the completed and approved ASR before samples can be received. If an ASR has not been received, the RSCC (or designated backup) notifies the Project Manager and assists him with information on how to complete and route the ASR.

b. The RSCC (or designated backup) performs the radioactive materials cooler/sample survey according to SOP 2420.15, Management of Radioactive Materials in the Laboratory Environment if the Purchasing and Receiving person (or designated backup) has not performed this task

c. The RSCC (or designated backup) notes the presence (or absence) and condition of custody tape seals on the outside of the cooler(s). A notation of this information is made in the file.

d. A completed COC (Attachment 2) must accompany each shipment of samples. The RSCC (or designated backup) requests that when a COC is not present, incomplete, or incorrect, that an amended or new one be submitted by the Project Manager or his designee.

e. The RSCC (or designated backup) removes all samples from the cooler(s) and notes the condition of each sample. Sample descriptions such as intact, broken, air bubbles present (for volatiles), or above 4 degrees Celsius (ice melted) are used to describe the sample condition. The sample condition is noted on the COC and field sheets (Attachment 4). Any additions or notes made on the COC or field sheets should be initialed and dated next to the correction or entry by the person making them. Any corrections should be done by placing a single line through the error and then noting the correct information. All corrections must be dated and signed by the person making the correction. Under no circumstances is information to be obliterated or whited out.

The Project Manager will be asked to make the necessary changes or corrections on the paperwork. In instances when the Project Manager is on site, he may designate the RSCC (or designated backup) to make minor changes for him. This may extend to changing a sample number or analysis listed, but does not include generating and completing all field paperwork. The RSCC (or designated backup) makes the indicated changes and sends the Project Manager a copy (no copy can be made of changes to sample tags on containers) of all paperwork changes. The Project Manager must respond back to the RSCC (or designated backup) as soon as possible if the corrections made are not as he intended. If no response is received within four working days, the RSCC (or designated backup) assumes that the Project Manager concurs.

f. If a sample is broken and there are no other containers from which the analysis can be done, the RSCC (or designated backup) initiates a memo of Non-Reportable Results (Attachment 5). This memo is sent to the Project Manager and the RLAB Data Coordinator to alert them to the fact that no data will be forthcoming for that sample/analysis. In addition, the RSCC (or designated backup) personally notifies the Project Manager of the sample loss so that the sample can be retaken at the Project Manager's

discretion. A notation is made on the COC to indicate any broken containers.

In the case of broken or leaking samples, all packing material (bubble or foam wrap, vermiculite, broken glass, etc.) and sample residue in the cooler are considered hazardous trash until proven otherwise through laboratory analysis of the samples.

g. The RSCC (or designated backup) verifies specific items on each of the samples and associated documents of the shipment. This includes determining if all documents/samples are consistent and accurate with regard to one another. It also includes identifying discrepancies in the use of EPA sample numbers. This circumstance usually occurs when the sampler adds additional unplanned samples. The Project Manager takes care of any necessary paperwork reconciliation to include assigning new EPA sample numbers if the ones identified are inappropriate.

(1) The RSCC (or designated backup) verifies the following specific information on the COC with respect to the sample container labels and field sheets:

(a) EPA Project/ASR number
(b) EPA sample numbers
(c) Number and type of sample containers
(d) Sample matrix

(2) The RSCC (or designated backup) verifies the following specific information on the sample containers with respect to the COC and field sheets:

(a) EPA Project/ASR number
(b) EPA sample numbers
(c) Type of containers
(d) Analysis
(e) Preservation

The RSCC (or designated backup) also observes the amount of sample received and determines if a sufficient quantity of sample was submitted to conduct the requested analyses. If an obviously insufficient sample was submitted, the RSCC (or designated backup) initiates a Notice of Non-Reportable Results form that is

forwarded to the Project Manager and Data Coordinator to inform them that no results will be forthcoming for that sample/analysis. A notation is also made on the COC. The RSCC (or designated backup) personally contacts the Project Manager and informs him of the situation so that he can retake the sample. In some instances, it is not determined until after the sample is in the laboratory that there is insufficient sample to conduct any or all of the tests requested. In these instances, RLAB informs the Project Manager as soon as possible of the existing situation. In some cases where some tests can be performed, but not all, the Project Manager determines the order and priority of tests for the sample.

(3) The RSCC (or designated backup) verifies the following specific information on the field sheets with respect to the COC and sample container labels:

(a) EPA Project/ASR number
(b) EPA sample numbers
(c) Matrix
(d) Container
(e) Analysis name
(f) Preservation

In addition to these items, the RSCC (or designated backup) notes any comments that may be inconsistent with the analysis requested (e.g., field filtered for total metals), information necessary to report the data (e.g., sample area or volume information for wipe and air samples), and if the field sheet is signed by the sampler in the space indicated.

h. When the information on the COC, field sheets, and sample tags is verified as complete and correct, the RSCC (or designated backup) checks the appropriate box at the bottom of the COC, "sealed" (custody taped) or "unsealed" (no custody tape present), and signs each COC and records the date and time of sample transfer. This action completes the sample receipt process and RLAB officially accepts custody of the samples. The yellow copy (or a photocopy) of the signed COC is given or mailed to the Project Manager. The original (white copy) is retained in the ENSV sample activity file maintained by RLAB.

2. Sample Log-In Procedure

a. From the ASR, the RSCC (or designated backup) identifies whether samples will be analyzed in-house (RLAB) or analyzed through the contract laboratory services (e.g., CLP or REAP). The samples are placed in the appropriate walk-in refrigerators located in the secured sample storage room (L-55) with the following exceptions: in-house volatile (VOA) samples are put in the refrigerator in the VOA analysis laboratory (L-30 for EPA and L-32 for ESAT, respectively). In-house samples requiring immediate analysis because of short holding times (Biological Oxygen Demand (BODs), chromium VI, or for other reasons, are delivered directly to the analyst by the RSCC (or designated backup).

b. The in-house sample receipt notification (SRN) is sent to RLAB personnel. The REAP and CLP sample receipt notification (Attachment 6) is only sent to the CATS staff. The information contained in the SRN includes ASR number, Project ID, due date (as assigned on the ASR by the RLAB Manager or designated person), the sample numbers, matrix, and the analyses requested. In the remarks area of each message type where the samples are stored, whether there will be more samples arriving, or not, who has the laboratory assignment, and the date when samples were collected. The sending of sample receipt notification instructions are outlined in section E.4 of this SOP.

(1) If the activity is being field shipped directly to a CLP or REAP laboratory, it is imperative that the Project Manager work closely with the RSCC (or designated backup). The Project Manager must return ENSV's copy of all paperwork (COC, field sheets, CLP shipment records, and/or REAP shipping documents) as soon as the samples are shipped to the CLP or REAP laboratory. The Project Manager works with the RSCC (or designated backup) to reconcile any discrepancies identified on the paperwork. The RSCC (or designated backup) logs the samples into R7LIMS when reconciliation of the paperwork is complete.

(2) The procedures for submitting ENSV's copy of the paperwork for samples sent directly to the REAP laboratory includes working closely with the REAP Project Officer (PO). Each Delivery Order must be coordinated and handled on an individual basis. The Project Manager or field personnel must fax ENSV's copy of all of the paperwork (COC, field sheets, and REAP shipping documents)

as soon as the samples are shipped to the REAP laboratory. The RSCC (or designated backup) and REAP PO (or designated backup) contacts the REAP laboratory informing them of the sample shipment.

c. The RSCC (or designated backup) makes the necessary entries into LIMS, reconciling the LIMS data with the sample paperwork and logs in the samples. Instructions for sample log-in are outlined in section E.3. of this SOP.

d. At the conclusion of all log-in activities for the shipment, the RSCC (or designated backup) promptly transmits the ENSV's copy of the field sheets and COC to the RLAB Data Coordinator. The Data Coordinator (or designated backup) files these documents according to the ASR number in the ENSV sample activity file maintained by RLAB.

e. If any hazardous trash is generated in the receipt of the samples, it is put in a polyethylene containment bag, tied, and labeled. The label includes the name of the sampling site, ASR number, and the date. The hazardous trash is turned over to the Sample Disposal Coordinator (or designated backup) for temporary storage in Sample Holding/Disposal (L-57).

f. The RSCC (or designated backup) places all uncontaminated used packing material from the cooler(s) in the dock-area trash cans, empties all water and ice from the cooler(s), and stacks the cleaned-up cooler(s) in the dock area. If there are directions to ship back specific cooler(s), the RSCC (or designated backup) coordinates their return.

3. Procedures for Sample Log-In Into R7LIMS

a. Confirm sample information (e.g., Project/ASR numbers, sample number(s))
b. Double click R7LIMS icon
c. Type Username and press enter
d. Type Password and press enter
e. Click once on Data Manager
f. Under ASR/Samples, double click Browse ASRs
g. Arrow to the ASR number and click to select
h. Click once on Log-In Samples
i. Type sample information from the field sheets

j. If the samples are assigned to a CLP laboratory, click once on Get SMO number. Confirm the next available CLP sample number on the list provided in cubicle 202B.
k. Click once on Log-In button
l. Check each sample to verify that the Received Date is the same for each sample (including the PE sample which has already been defined in the system). The computer uses the received date to create each particular F2L download file
m. Check the sample container list, and edit as necessary, to assure that it matches the COC
n. Click Exit when all samples are logged in

4. Instructions for Sample Receipt Notification to Regional Laboratory Personnel

a. Double click R7LIMS icon
b. Type Username and press enter
c. Type Password and press enter
d. Click once on Data Manager
e. Under ASRs/Samples, double click Browse ASRs
f. Using the mouse, click once and highlight the ASR number
g. Click once on SRN button
h. At lab, arrow down to Receipt Date tab needed
i. Under Report Sample, arrow down to Numbers tab
j. Click once in the Comments box and type the following information in the Comments section:
   (1) The lab for which the samples are assigned (e.g., REAP, CLP, EPA, or ESAT);
   (2) The refrigerators or freezers where the samples will be stored for in-house analyses (e.g., 1st or 2nd walk-in refrigerators in L-55, EPA volatiles refrigerator in L-30 and the ESAT volatiles refrigerator in L-32)
   (3) Whether this SRN set completes or does not complete the specific ASR.
k. Click once on Generate Receipt
l. The computer will pull up an on-screen preview of the SRN in Acrobat Adobe Reader
m. Click once on Email
n. The computer will pull up an Email Address Screen
o. Click once on Addresses
p. The computer will pull up a Select Email To Information screen

q. Select the appropriate RLAB staff to receive the SRN by clicking once on the appropriate staff member
r. Click once on the single arrow
s. Repeat until all appropriate personnel have been selected
t. Click once on Close
u. Click once in the Subject box and type lab assignment based on the information contained in the ASR, (e.g., EPA/REAP SRN -ASR 2199(not)complete)
v. Click once on Store SRN and send Email
w. A message box will be generated and state that the Email was sent.
x. Click once on OK
y. The computer will pull up a screen that will show the number of SRNs sent for that ASR. Click once on Close
z. An Acrobat Report Viewer screen will appear. Click once on Close
aa. A Sample Receipt Notice screen will appear. Click once on Exit.

5. Instructions for Generation of the COC Record for REAP

a. The RSCC (or designated back-up) or ESAT contractor will receive the COC and field sheets with the sample shipment. The information on these forms is compared to the condition and actual samples in the shipment to verify that the information is recorded correctly.

b. The RSCC (or designated backup) or ESAT contractor takes the shipment paperwork (Attachment 3) and records the sample information onto an EPA COC Record (FORM-7-EPA-9262). The RSCC (or designated backup) or ESAT contractor records the Task Order Number, EPA Reference Number, and name of the REAP laboratory at the top of the EPA COC Record. The Project Manager, ASR/Project ID, Sample Collection Date and number of pages are recorded in the appropriate boxes. Information pertaining to the number and type of sample containers, media and QC codes are recorded for each sample number on this form. The total amount of samples and containers and number of ice chests is recorded in the block labeled Description of Shipment. The shipment carrier and shipping document number is noted in the block labeled Mode of Shipment. The RSCC (or designated backup) or ESAT contractor must sign the COC and note the date and time that the shipment is to be relinquished to the sample carrier. The RSCC (or designated backup) must check mark whether the shipment has been sealed or unsealed. The white copy is placed with the shipment paperwork which is

sealed in a document holder attached to the inside lid of the cooler. The yellow carbon copy is placed in the RSCC's in-box.

6. Instructions for the Creation of F2L files for CLP Shipments

a. Double click on R7LIMS icon
b. Type Username and press enter
c. Type password and press enter
d. Click once on Data Manager
e. Under Samples Results, double click Single ASR Criteria
f. Click once on All
g. Click once on Browse Single ASR
h. Click and highlight the particular ASR to be downloaded to an F2L file.
i. Click once on the F2L Download. The computer will pull up a screen reading Download Forms2Lite Site File.
j. At Sample, click Received
k. Click CLP
l. At Date Received, click once on the log-in date for the needed shipment ("control-click" can be used if multiple dates are needed)
m. Click once on the Select File at Output File
n. At Output File, type k:\ASR#F2Ldwnld.F2L (e.g., k:\2127F2Ldwnld.F2L)
o. Click once on Download. A message will appear stating that the file has been downloaded to k:\ASR#F2Ldwnld.F2L (e.g., k:\2127F2Ldwnld.F2L)
p. Click once on OK
q. Click once on Exit to return to Browse Single ASR Results
r. Click once on Exit to return to Browse Main Menu
s. Click on File to exit R7LIMS
t. Double click F2Lite5.1 icon
u. A Forms2Lite5.1 box will appear. Click once on Cancel
v. Click once on File at the upper left hand corner of the screen
w. Click once on Import/Export
x. Click once on Import a F2L.5.x file
y. Click once on Next
z. Click once on Browse on Select File: Screen on the Import Export Wizard. Select date to Import (Make sure that the Import Measurements Data and the Import Archived TR Date do not have a check mark)
aa. Click once on arrow at Look in:
bb. Arrow down to k:\ drive
cc. Click once on file to be imported (e.g., 2127F2Ldwnld.F2L)
dd. The appropriate ASR to be downloaded will appear in File Name
ee. Click once on Open

ff. Click once on Finish
gg. Click once on OK after import has been completed
hh. Click once on File in upper left hand corner
ii. Click once on Exit to exit from Forms2Lite5.1

7. Instructions for the Generation of the Traffic Report (Attachment 7) and Sample Labels in Forms2Lite5.1

a. Click once on File
b. Click once on Open Site
c. Arrow to the downloaded Site and click to select
d. The computer will pull up a screen stating Step One: Site Information-(Site Name)
e. The information from the site should already be downloaded into the system with the exception of the Case Number. Type in the case number
f. Click Next
g. The computer will pull up a screen stating Step Two: Select Sampling Team
h. Click Next
i. Step Three: Select Analysis. The particular analysis should already be downloaded into the system
j. Click Next
k. The computer will pull up Step Four: Station/Location Information. The information should already be in the system except for the Concentration. At Concentration click the down arrow and select the appropriate concentration used in the field sheets for the site
l. Click Next
m. The computer will pull up Step Five: Assign Bottles. In the Select Station/Location, use the down arrow to select the sample(s) to be assigned Quality Control samples. The sample number(s) should appear in the Assigned Analysis With Sample Number table
n. Click Lab QC Type.
o. Click the down arrow and click the appropriate QC type
p. Click Next. A Forms II Lite query will appear to verify that QC has been assigned
q. Click OK
r. Click Next
s. Step Six: Assign Lab
t. Click down arrow at Lab Code
u. Click appropriate lab for a particular analysis(es) according to the assignment in the RSCC's LIMS ASRs & Sample Receipt notebook

v. All samples for that particular lab should appear in the Assigned Samples to Labs table
w. Repeat this procedure until all samples are assigned to the appropriate laboratories
x. Click Next
y. Step Seven: Assign Carrier
z. Click down arrow at Carrier
aa. Click the appropriate Carrier
bb. Type in the Date Shipped
cc. Type in the Airbill number
dd. Click the Lab header at the top of the table to sort by a particular lab when shipments are going to more than one lab
ee. Click and highlight the samples going to a particular lab
ff. Click Assign
gg. Repeat this procedure for each airbill and its associated analyses/lab until all samples are assigned
hh. Click Finish
ii. The computer will pull up Print/View a Specific TR screen
jj. Verify whether the shipment is complete or not complete from the COC delivered from the field. If the shipment is complete, double click the Complete box and query will change from No to Yes
kk. At the Select a View, click Lab Copy
ll. Click Print (this copy is retained to be shipped with the samples). Sign and date the document(s) in the section marked Relinquished By and initial for the Sampler in the Sampler Signature section
mm. Click Region Copy
nn. Click Print (this copy is given to the RSCC or designated backup). Sign and date the document(s) in the section marked Relinquished By and initial for the Sampler in the Sampler Signature section.
oo. Click Back until Step Five: Assign Bottles screen
pp. Click Generate Labels
qq. Click All Samples for Site
rr. Click Generate Label
ss. Click Edit Label
tt. Arrow to appropriate Avery number that matches the dimensions of the label to be used for the sample tags
uu. Click Next
vv. Verify that the Font Name is Arial
ww. Verify that the Font Weight is Normal
xx. Verify that the Font Size is 10
yy. Verify that the Text Color is black

zz. Click Next
aaa. Verify that the prototype label information is correct
bbb. Click Next
ccc. The computer will pull up the query, "What name would you like for your label?"
ddd. Type the string ASR#initialsshipmentdate (without spaces, e.g., 2171lh1024)
eee. Click Finish
fff. A preview of the sample tags will appear. Verify that the sample information is correct
ggg. Click Print
hhh. Select the printer designated for sample tag generation
iii. Click Print
jjj. Verify that the sample tag information is correct
kkk. Click the X box at the top right-hand corner of the screen
lll. Click Close
mmm. The computer will query, "Would you like to exit this Site?" Click Yes

8. Procedures to Export an F2L Site File and E-mail Instructions

a. Click on File at the upper left-hand corner of the screen
b. Click Import/Export
c. Click to highlight Export to a File at the Import Export Wizard screen
d. Click Next
e. Arrow to Site and click to highlight
f. Click Finish
g. At File Name, type the following string without spaces: R7ASR#Case#initials(not)complete.F2L, (e.g., R721723125lhnotcomplete.F2L)
h. Click Save
i. A computer message will appear stating that the file has been exported
j. Click OK
k. Click File
l. Click Exit
m. The computer will exit Forms2Lite
n. Double click LOTUS Notes icon
o. Type in password and press enter
p. Click OK
q. Click Mail
r. Click New Memo

s. Type in the names of the RSCC, CLP Program Officer and the Sample Management Office coordinator
t. Press tab and type the following information in the Subject line: R7case#ASR#complete.F2L or R7case#ASR#notcomplete.F2L (without spaces) e.g., R731292202notcomplete.F2L
u. Press tab until the cursor is in the body of the message
v. Click File
w. Click Attach
x. Type: k:\ and press Enter
y. Choose the file on the k:\ drive
z. Click Send
aa. Exit LOTUS NOTES

F. QUALITY ASSURANCE AND QUALITY CONTROL

All sample information is verified as correct through comparison of the field sheets, sample tags, and shipping documentation (such as, traffic reports, and/or COC). All documents must be consistent prior to acceptance and properly maintained throughout the shipping and analytical data files process. Any discrepancies are resolved through consultation with the Project Manager or RSCC (or designated back-up).

G. REFERENCES

1. US EPA, Region 7, "RLAB Analytical Data Management Procedures" Environmental Services Division Operations and Quality Assurance Manual, SOP 2410.1

2. USEPA, Region 7, "Management of Radioactive Materials in the Laboratory Environment", SOP 2420.15

SOP No. 2420.1E

Attachment 1

US EPA Region 7 Analytical Services Request (ASR)

Project ID: ASR Number: Projected Delivery Date:

Project Dest:
City: State: Program:
Site Name: Site OU
Site ID: CERCLIS ID:
GPRA PRC:

Project Manager:
Organization: Phone Number:

Contact:
Organization: Contact Phone:

ASR Purpose:
Comments:

Is this activity currently or potentially a criminal investigation?
Has a QAPP for the requested services been approved?

For health, safety and environmental compliance are any samples expected to contain:

Dioxin > 1ppb:
RCRA Listed Wastes:
Toxic/Hazardous Chemicals >1000ppm:

No. of Samples	Reg No	CNS List	Conc of Interest	Expected Conc

Special Analytical Requirements or Comments:

Date Submitted: By: ASR Status:
Date Accepted: By: RLAB Turn Around Time: Days
Date Transmitted: By: ANOP Turn Around Time: Days

Attachment 2

CHAIN OF CUSTODY RECORD
ENVIRONMENTAL PROTECTION AGENCY REGION VII

ACTIVITY LEADER(Print)	NAME OF SURVEY OR ACTIVITY	DATE OF COLLECTION (DAY / MONTH / YEAR)	SHEET 01

CONTENTS OF SHIPMENT

SAMPLE NUMBER	TYPE OF CONTAINERS: CUBITAINER	BOTTLE	BOTTLE	BOTTLE	VOA SET (2 VIALS EA)	SAMPLED MEDIA: water	soil	sediment	sludge	other	RECEIVING LABORATORY REMARKS/OTHER INFORMATION (condition of samples upon receipt, other sample numbers, etc.)
	NUMBERS OF CONTAINERS PER SAMPLE NUMBER										

DESCRIPTION OF SHIPMENT	MODE OF SHIPMENT
_____ PIECE(S) CONSISTING OF _____ BOX(ES)	_____ COMMERCIAL CARRIER: _____
_____ ICE CHEST(S); OTHER _____	_____ COURIER
	_____ SAMPLER CONVEYED (SHIPPING DOCUMENT NUMBER) _____

PERSONNEL CUSTODY RECORD

RELINQUISHED BY (SAMPLER)	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	

SOP No. 2420.1E

Attachment 3

REAP SHIPPING DOCUMENT
(ARDL)

SHIPPING INFORMATION

TASK ORDER#: 0154 SHIPMENT DATE: 08/20/2003

EPA REFERENCE#: 2111A03 SHIPMENT NO.: 1

SAMPLE INFORMATION

SAMPLE #	MATRIX	PARAMETERS	DATE COLLECTED
2111-1	Water	Explosives	08/19/2003
2111-2	Water	Explosives	08/19/2003

REMARKS:

Attachment 4

Sample Collection Field Sheet

U.S. EPA Region VII
Kansas City, KS

ASR Number: Sample Number: QC Code: Matrix: Tag ID:

Project ID: Project Manager:
Project Desc:
City: State:
Program:
Site Name: Site ID: Site OU:

Location Desc: _____

External Sample Number: _____

Expected Conc: (or circle one: Low Medium High) Date Time (24 Hr)

Latitude: ___ ___ ___ Sample Collection: Start ___/___/___ ____:____
Longitude: ___ ___ ___ End ___/___/___ ____:____

Laboratory Analyses:

Container	Preservative	Holding Time	Analysis
1 - 1 Liter Cubitainer	HNO3 acidify, 4 Deg C	180 Days	Metals in Water by ICP

Sample Comments:

Sample Collected By: _____

SOP No. 2420.1E

Attachment 5

Date

MEMORANDUM

SUBJECT: Notice of Non-Reportable Sample Results

FROM:

TO: Primary Data File ASR Number: _____/Site Name: _____)

Data will not be reported for the following samples:

Sample Number	Parameter	Reason

cc: EPA Project Manager

Attachment 6

Sample Receipt Notice

11/07/2003 10:52

ASR Number: 2216
Samples Received: 11/07/2003
RLAB T-A-T: 30
Criminal: No
Project Id: WGP99
Project Desc: Webster City WWTF

Lab: (All)
Report Sample: Numbers

Req No	Analysis	Matrix	Analyst Lab	Pri	Sec	Samples
1	NH3-N W	Water	EPA	DAD		6-__
1	Met Water	Water	EPA		DAD	6-__

Comments:

EPA USEPA Contract Laboratory Program
Organic Traffic Report & Chain of Custody Record

Case No: 31795 R
DAS No:

Region:	7	Date Shipped:	6/5/2003	Chain of Custody Record	Sampler Signature:
Project Code:	RS07WC/2054	Carrier Name:	FedEx	Relinquished By (Date / Time)	Received By (Date / Time)
Account Code:	50102D	Airbill:	841407300500	1 6/5/03	
CERCLIS ID:	IAD984601039	Shipped to:	Datachem Laboratories, Inc. 960 West LeVoy Drive Salt Lake City UT 84123 (801) 266-7700	2	
Spill ID:	07WC			3	
Site Name/State:	Alberty City SBA sampling/IA			4	
Project Leader:	Jeff Pritchard				
Action:	Remedial Action				
Sampling Co:	Tetra-Tech. EMI				

ORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	INORGANIC SAMPLE No.	QC Type
G0HA8	Municipal Water Supply/ Jeff Pritchard	L/G	LDL VOA (21)	20544525 (HCL), 20544526 (HCL), 20544527 (HCL), 20544528 (HCL) (4)	2054-101/MW#2	S: 6/3/2003 8:40		-
G0HA9	Municipal Water Supply/ Jeff Pritchard	L/G	LDL VOA (21)	20544529 (HCL), 20544530 (HCL), 20544531 (HCL), 20544532 (HCL) (4)	2054-102/MW#3	S: 6/3/2003 8:52		-
G0HB0	Municipal Water Supply/ Jeff Pritchard	L/G	LDL VOA (21)	20544533 (HCL), 20544534 (HCL), 20544535 (HCL), 20544536 (HCL) (4)	2054-103/MW-9B	S: 6/3/2003 10:45		-
G0HB1	Surface Water/ Jeff Pritchard	L/G	LDL VOA (21)	20544537 (HCL), 20544538 (HCL), 20544539 (HCL), 20544540 (HCL) (4)	2054-104/FSKS	S: 6/3/2003 11:06		-
G0HB2	Municipal Water Supply/ Jeff Pritchard	L/G	LDL VOA (21)	20544541 (HCL), 20544542 (HCL), 20544543 (HCL), 20544544 (HCL) (4)	54-105FB/LDL VOA Trip BLS:	6/3/2003 11:20		Trip Blank
G0HB3	PE Water/ Jeff Pritchard	L/G	LDL VOA (21)	20544545 (HCL), 20544546 (HCL), 20544547 (HCL), 20544548 (HCL) (4)	2054-106/CLP PE sampleS:	6/3/2003 8:35		PE

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC: G0HA8	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key:	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____
LDL VOA = Low Con.VOA(OLC03.2)			

TR Number: 7-304918220-060503-0001

REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, 2000 Edmund Halley Dr., Reston, VA. 20191-3400 Phone 703/264-8348 Fax 703/264-8222

STANDARD OPERATING PROCEDURE

No. 2430.6C

PERIODIC INTERNAL PROGRAM REVIEW OF THE REGION 7 LABORATORY

December 15, 2003

Harold Brown
ENSV/RLAB/CATS

APPROVED:

Peer Reviewer — Date: 12-18-03

Laboratory Manager — Date: 19 Dec 03

Harold D. Brown — Date: 12/19/03

Independent QA Reviewer

Recertified:

Reviewer	HBB				
Date	10/04/05				

Table of Contents

A. PURPOSE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to establish a uniform process for performing periodic internal program reviews of the EPA Region 7 Laboratory (RLAB). The policies and procedures contained in this SOP are applicable to all EPA and contractor personnel, and are to be used when performing periodic internal program reviews of RLAB.

B. POLICY

It shall be the policy of RLAB to periodically perform internal program reviews for the purpose of determining if the laboratory program is being operated in substantial conformance with applicable laws, regulations, regional policies, internal laboratory policies, and SOPs.

C. DEFINITIONS

1. NELAC - National Environmental Laboratory Accreditation Conference
2. OIRM - Office of Information Resources Management
3. FMFIA - Federal Managers' Financial Integrity Act
4. FAR - Federal Acquisition Regulations
5. EPAAR - EPA Acquisition Regulations

D. PROCEDURES

1. Frequency of Internal Review

   The goal is to complete at least one internal laboratory program review per 12-month period. Reviews may be conducted more frequently if it is determined by RLAB management that there is a need for review in one or more specific areas.

2. Scope of the Review

   a) When performing an internal review, the review may include any or all of the areas identified in "b" below. This means that a given review might cover only one area, or all of them, depending on the specific goals of the review. The determination of what areas are to be reviewed will be made jointly by RLAB management (RLAB Director, Independent QA Reviewer, Analytical Operations Program Manager, and Contract Analysis and Technical Support Program Manager) prior to the initiation of the review.

b) The following is a listing of POTENTIAL areas for inclusion in an internal review. A reference is provided for each area on the list. The list is not necessarily exhaustive since other areas of interest may be added if desired by RLAB management.

1) General Laboratory Practices; REFERENCE: NELAC Standards, Chapter 5, Appendix D, Section D.1.6

2) Lab Equipment and Instrumentation; REFERENCE: NELAC Standards, Chapter 5, Section 5.8

3) Personnel; REFERENCE: NELAC Standards, Chapter 5, Section 5.6

4) Facility; REFERENCE: NELAC Standards, Chapter 5, Section 5.7

5) Safety; REFERENCE: SOP # 2440.3, "Containment Laboratory Safety Plan;" "Chemical Hygiene Plan"

6) QC Spot Check Requirements; REFERENCE: SOP # 2430.5, "Quality Control Spot Checks of Regional Laboratory Data Packages"

7) Laboratory Management Plans; REFERENCE: SOP # 2440.5 "U.S. EPA Region 7 Laboratory Quality Assurance Operating Plan," Section 13.2 – "Management Systems Evaluation"

8) Laboratory Quality Assurance Operating Plan; REFERENCE: SOP # 2440.5, "U.S. EPA Region 7 Laboratory Quality Assurance Operating Plan"

9) Good Automated Laboratory Practices (GALP); REFERENCE: EPA OIRM Document 2185 – GALP

10) Management Controls; REFERENCE: FMFIA Guidance

11) Contract Practices; REFERENCE: FAR/EPAAR

3. Conduct of the Review

a) A person or persons appointed by RLAB management will perform the internal review. The reviewers will be selected for their expertise in the areas to be checked and will, as much as is practical, have had no connection to or personal interest in the area to be reviewed.

b) All pertinent data related to the area will be examined to determine that proper procedures are being followed. The following is a general description of each of the potential areas which may be selected for review:

1) General Laboratory Practices - Conformance with currently applicable NELAC Standards for this area will be checked. See the reference in Section 2 above.

2) Lab Equipment & Instrumentation - Conformance with currently applicable NELAC Standards for this area will be checked. See the reference in Section 2 above.

3) Personnel - Conformance with currently applicable NELAC Standards for this area will be checked. See the reference in Section 2 above.

4) Facility - Conformance with currently applicable NELAC Standards for this area will be checked. See the reference in Section 2 above.

5) Safety - The laboratory's conformance to SOP # 2440.3, "Containment Laboratory Safety Plan," and "Chemical Hygiene Plan," will be evaluated.

6) QC Spot Check Requirements - Conformance with SOP # 2430.5, "Quality Control Spot Checks of Regional Laboratory Data Packages," will be evaluated.

7) Laboratory Management Plans - Conformance with the requirements of SOP # 2440.5, "U.S. EPA Region 7 Laboratory Quality Assurance Operating Plan," Section 13.2 – "Management Systems Evaluation," will be evaluated. This includes whether the annual evaluation has been performed and if the evaluation

substantially meets the SOP requirements.

8) Laboratory Quality Assurance Operating Plan (QAOP) - An evaluation of overall conformance with the SOP # 2440.5 "U.S. EPA Region 7 Laboratory Quality Assurance Operating Plan" will be conducted, including an evaluation of the adequacy of the QAOP itself and its need for being updated.

9) Good Automated Laboratory Practices - This check will verify that automated data management practices as recommended and described in "Good Automated Laboratory Practices," EPA OIRM Document 2185, August 1995, are substantially followed.

10) Management Controls - The laboratory's conformance with current FMFIA guidance will be assessed.

11) Contract Practices – RLAB's conformance with contract practices as described in the FAR and EPAAR will be assessed.

4. Reports/Responses/Corrective Actions

a) The RLAB Independent QA Reviewer will coordinate the preparation of reports detailing the findings of the internal review. These reports will summarize the findings and make recommendations as appropriate. Prior to the report being finalized, draft copies will be furnished to RLAB management (see C.2.a. above) for review and comment. After this review and comment process, the Independent QA Reviewer will prepare the final report and distribute it to RLAB management.

b) RLAB management will review the report and take actions, as appropriate, in response to findings and recommendations applicable to their areas of responsibility.

c) If the internal review reveals the need for formal corrective actions, they will be planned, implemented, and tracked as described in the RLAB QAOP.

E. REFERENCES

1. NELAC Standards
2. SOP # 2440.3, "Containment Laboratory Safety Plan"
3. "Chemical Hygiene Plan"
4. SOP # 2430.5, "Quality Control Spot Checks of Regional Laboratory Data Packages"
5. SOP # 2440.5, "U.S. EPA Region 7 Laboratory Quality Assurance Operating Plan"
6. EPA OIRM Document 2185
7. FMFIA Guidance
8. FAR
9. EPAAR

Environmental Protection Agency

901 N 5th Street
Kansas City, KS 66101

MEMORANDUM

DATE: August 25, 2006

SUBJECT: Addendum to the U.S. EPA Region 7 SOP 2430.6C, Periodic Internal Program Review of the Region 7 Laboratory

FROM: Harold Brown, Independent QA Reviewer, ENSV/RLAB HSB

THRU: Harry Kimball, ENSV/RLAB HK
Dale Bates, Laboratory Branch Chief, ENSV/RLAB DWB

TO: All RLAB and ESAT Contractor Staff
Method Files: SOP 2430.6C

This addendum provides updated information on the RLAB policy for conducting internal technical program reviews as described in SOP 2430.6C.

The following requirements are hereby incorporated into Section D, Procedures, of the subject SOP.

1. All technical areas will be included in the internal review.
2. Data produced by laboratory personnel will be reviewed following the procedures described in SOPs 2410.10 and 2430.5.
3. Interviews with the laboratory personnel producing data will be an integral part of the internal review process.
4. The review of each technical area will be documented by using detailed checklists.
5. Internal review will be conducted by qualified personnel who are independent of the data generation process.
6. A comprehensive Internal Review Report will be submitted to the Independent QA Reviewer (Laboratory QAO) and the Laboratory Manager. The report will include a summary of findings, proposed corrective actions, recommendations for quality improvement, and supporting documentation (i.e., checklists, notes, etc.).
7. The Internal Review Report will be a primary reference for the Management Systems Review.

STANDARD OPERATING PROCEDURE

No. 2430.12E

REGIONAL LABORATORY QUALITY CONTROL POLICY

December 18, 2003

by

Harry Kimball

ENSV/RLAB

APPROVED:

Leslye E Werner — Peer Reviewer — 12/23/03 Date

Joe Anderson for — Manager, Regional Laboratory — 12/24/03 Date

Harold D. Brown — Independent QA Reviewer — 12/24/03 Date

Recertified

Reviewer	HK	HK			
Date	11-19-04	1-5-07			

Table of Contents

Attachments

A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to document the established Quality Control (QC) Policy for Region 7 Laboratory in-house analytical operations. This policy contains a series of uniform QC procedures to ensure that data are generated with sufficient and appropriate controls to meet client requirements for data quality. This SOP is intended to contain sufficient versatility and generality to cover changing client needs and program focus. The reader is referred to SOP 2410.10, "Analytical Data Submission Package Contents and Review" and SOP 2410.15, "Estimating and Documenting Data Quality," for additional details on the implementation of the policies described herein. It is recognized that some analyses cannot conform to all conditions in this policy. The QC requirements specified in particular regulations, reference methods, and/or RLAB Methods shall be followed.

The policies and procedures detailed in this SOP shall be utilized by Region 7 Laboratory personnel and in-house contractor personnel responsible for implementing, performing, reviewing, and/or reporting analyses. These policies and procedures shall be considered minimum requirements. Additional procedures may also be required by regulatory, method, contract, or SOP references. The QC policies and procedures required of contract laboratories [Contract Laboratory Program (CLP) and Regional Environmental Analysis Program (REAP) Contract] may be different than those listed below. Whenever possible, contract laboratories shall be required to follow the procedures and meet the standards listed below. However, where a contract Statement of Work (SOW) is incompatible with these procedures and standards, the QC requirements of the contract SOW will take precedence.

It is the goal of the Region 7 Laboratory to produce legally- and technically-defensible data of known and documented quality, which is useable for the purpose(s) to which it was intended. Every effort will be made to achieve this goal.

B. POLICY

The Region 7 analytical QC program will establish, maintain, and monitor data quality with internal QC checks. Internal QC checks shall be used to address three questions:

1. Has the laboratory's ability to produce data of acceptable quality been established and maintained?

2. Were laboratory operations "in control" (operating within acceptable QC limitations) during data generation?

3. What effect did the sample matrix have on the data being generated?

The first question is addressed by initial and on-going demonstrations of capability, establishment of method detection limits, and preventive maintenance procedures and records. The laboratory's overall abilities are assessed by the performance and documentation of these initial and on-going QC measures.

The second question is addressed by calibration checks, positive and negative method performance checks, and by following sample preservation and maximum holding-time protocols. Method calibration and positive performance checks are compared to established control limits. This information, in conjunction with negative performance checks (blanks), is used to assess analytical batch-level laboratory performance.

The third question is addressed by matrix-specific QC. Matrix-specific QC is based on the use of actual environmental samples for precision and bias determinations and relies on the analysis of matrix spike samples. Matrix spike information is used to assess the effect of the sample matrix on analytical data.

To meet the laboratory's quality objectives, the following steps will be taken:

1. All data generated within or for the Region 7 Laboratory will be subject to QC review.

2. Initial and ongoing demonstration of capability and determination of method detection limits shall be performed and documented for each analysis (RLAB Method) performed routinely in the laboratory.

3. One complete set of QC checks (as appropriate to each method) shall be run with each batch of field samples. A batch shall consist of no more than 20 field samples. (For the purposes of defining a batch, field samples include: "real" field samples, field duplicates, field blanks, field spikes, performance testing, and blind performance evaluation samples.) In general, a "complete set of QC checks" will include, depending on the method, at least these four analyses:

   - Laboratory control sample
   - Method blank
   - Matrix spike
   - Matrix spike duplicate

   The CLP SOWs specify in detail what QC checks will be run and when. Each Task Order issued under the REAP Contract shall specify what QC checks are to be run and how often.

4. For each analytical measurement performed routinely in the laboratory, QC information shall be stored in the laboratory information management system. From this historical database, control limits shall be established for each routine test measurement. Control limits shall be updated at least every two years in conjunction with the review of the corresponding RLAB Method. (See SOP 2410.15 for control limit calculation procedures.)

5. The Region 7 Laboratory shall report all in-house generated results, rounded to the number of significant figures specified in the RLAB Method (see SOP 2410.19, "Significant Figures (Digits)"), down to the Region 7 Reporting Limit (RL). RLAB Method RL's must be greater than the method detection limit (MDL) and are, typically, approximately three times the MDL. The CLP Statements of Work (SOW) specify "Contract Required Quantitation Limits (CRQLs)," down to which all CLP data will be reported. Each REAP Task Order SOW shall specify the limits to be reported (RLs or CRQLs), based on the project data quality objectives.

6. For conducting analyses in-house (not by an out-source contract laboratory), if any QC check fails to meet applicable control limits, corrective action is required for all associated data (e.g. the same prep and/or analysis batch). This includes a review of sample preparation and instrument performance data and/or a consultation with the supervisor to discuss data qualification and/or sample re-analysis. Possible courses of action may include, but are not limited to, dilution, re-analysis, method of standard additions, or use of a different method. It is possible that the outlier may be due to a challenging matrix or may be beyond the control of the laboratory, in which case no corrective action can be taken. The analytical supervisor is responsible for establishing and documenting the policy for common corrective actions. The analyst should consult the supervisor whenever there is any question about the appropriate corrective action for a given sample analysis. All corrective actions shall be fully documented in the back-up data file. When data quality exceptions are not corrected, associated data will be qualified (see SOP 2410.10 for data qualification guidelines).

   For conducting analyses under the CLP SOWs, the procedures for dealing with QC check anomalies are detailed in the SOWs and in SOPs 2430.2, 2430.3, and 2430.4. For conducting analyses under the REAP Contract, the REAP laboratory shall follow technically sound procedures for dealing with QC check anomalies and fully document all anomalies, corrective actions and their results in the data deliverable.

7. Any given procedure or activity may include additional QC which can be defined separately as the need arises.

C. DEFINITIONS

Analytical Services Request (ASR). An ASR is a document used by a Project Manager to request analytical services from RLAB. On the ASR, the Project Manager explicitly states the matrix of the samples to be collected, the analytical method to be applied to those samples, the analytes for which results are required, and any additional data quality objectives. Implicit in this request is that reported analytical results meet the requirements of this QC Policy.

Method Detection Limit (MDL). This term refers to the quantitative expression for the minimum concentration of a substance that an analytical method is capable of detecting and quantifying with 99% confidence that the concentration of the substance is greater than zero. The method detection limit is determined from the analysis of replicate samples in accordance with the procedures outlined in Title 40, Code of Federal Regulations (CFR), Part 136 Appendix B or as specified in the method.

Reporting Limit (RL). The Reporting Limit for an analyte is the concentration represented by the lowest level in the initial calibration curve where the analyte is detected, unless otherwise specified in the RLAB Method. RL's are, typically, approximately three times the method detection limit (MDL).

RLAB. The EPA Region 7 Laboratory.

RLAB Approved Data. Data that have been through the laboratory's data verification process (verified data). Any data that do not meet all prescribed requirements during the data verification process are qualified and accompanied by a narrative explaining the nature of the data quality exception. Data in the Data Transmittal sent by RLAB to the Project Manager are RLAB Approved Data.

RLAB Method. The RLAB Method is the Region 7 Laboratory document that describes an analytical procedure. Typically these are based on published EPA methods.

Validated Data. Data that have been reviewed by the Project Manager, or other end user, for conformance to the requirements of the specific use for which the data were generated. Typically, the first step in this process is for the Project Manager to obtain RLAB Approved Data from the Region 7 Laboratory.

Verified Data. Data that have been reviewed by RLAB for conformance to the requirements specified, implicitly and explicitly, in the analytical services request. These include the requirements set forth in this SOP (and SOPs referenced by this SOP), specific quality control requirements of the requested analysis (contained in the RLAB Method), and any additional requirements specified by the Project Manager in the ASR.

D. PERSONNEL RESPONSIBILITIES AND AUTHORITY

1. Laboratory Management

a. Members

The supervisors and managers who direct the analytical work of each laboratory group are directly responsible for ensuring that all employees reporting to them are complying with the Regional Laboratory QC Policy.

b. Responsibilities

Laboratory management is responsible for:

(1) Actively supporting the implementation of the Regional Laboratory QC Policy within the laboratory

(2) Maintaining accurate SOPs, SLOMs, and RLAB Methods and enforcing their use in the laboratory

(3) Maintaining a work environment that emphasizes the importance of data quality

(4) Providing management support to the Regional QA Manager

c. Authority

The managers and supervisors of the laboratory have the authority to accept or reject data based on well-defined QC criteria. In addition, managers and supervisors can accept data which fall outside of normal QC limits if, in their judgment, there are technical reasons which warrant the acceptance of the data. These circumstances must be well documented and any needed corrective action identified by the incident must be defined and initiated. The analyst shall generate this documentation and the supervisor shall approve the documentation. All such documentation shall

be maintained in the primary data file. The authority of the laboratory management comes directly from the ENSV Division Director.

2. Laboratory Personnel

a. Members

All laboratory personnel involved in the generation and reporting of data have a responsibility to understand and follow the Regional Laboratory QC Policy.

b. Responsibilities

Laboratory personnel are responsible for:

(1) Possessing a working knowledge of the Regional Laboratory QC Policy

(2) Ensuring that all work is generated in compliance with the Regional Laboratory QC Policy

(1) Performing all work according to written SOPs, SLOMs, and RLAB Methods

(2) Ensuring that all documentation related to their work is complete and accurate

(3) Providing management with immediate notification of quality problems

c. Authority

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC acceptance criteria. The acceptance of data which fall outside QC criteria must be approved by laboratory management. The authority of the laboratory personnel flows from the Laboratory Program Managers.

3. In-House Contractors

The requirements discussed above as applicable to EPA employees shall also be required of all contractor employees using Regional Laboratory equipment and facilities.

E. SPECIFIC ELEMENTS OF THE LABORATORY QC POLICY

1. Initial and On-going Demonstration of Capability

a. Initial and on-going demonstration of capability is important for the documentation of the laboratory's ability to produce data of acceptable quality initially and on a continuing basis. This process is fully described in SOP 2410.11, "Analytical Proficiency Demonstration in the Region 7 Laboratory."

b. Contract laboratories conduct initial and ongoing QC as a part of obtaining the contracts, and in accordance with SOW and Task Order requirements. Such QC shall be checked as a part of the routine on-site evaluation process for contract laboratories.

2. Method Detection Limits

a. Method detection limits will be determined by the procedure specified in the method or by the 7 to 10 replicate procedure described in 40 CFR Part 136 Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit."

b. The sample preparation, analysis, and calculation procedures used for field sample analysis will be employed in the determination of the method detection limit.

c. A method detection limit study will be performed initially when a new method is implemented and subsequently whenever there is a significant change in the method or instrumentation such that the detection limit may be affected.

3. Preventative Maintenance

Analytical instrumentation and equipment will be maintained by following established preventative maintenance procedures (per manufacturer's recommendations) and records of such maintenance shall be kept.

4. Calibration Data

a. Standard/Reagent Preparation

A critical element in the generation of quality data, is the purity/quality and traceability of the standard solutions and reagents used in the analytical operations. To ensure the highest purity possible, all primary reference standards and standard solutions are obtained from the National Institute of Standards and Technology (NIST), the EPA Repository, or other reliable commercial sources (e.g. NIST traceable).

Standard solutions are validated prior to use. Validation procedures can range from a check for chromatographic purity to verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source and should be specified in the RLAB Method. Care is exercised in the proper storage and handling of standard solutions, and all containers are labeled as to compound, concentration, solvent, expiration date, initials of preparer and preparation date. Stock and working standards are checked regularly for expiration and signs of deterioration such as discoloration, formation of precipitates, or change of concentration. Supervisors are responsible for establishing policies for determining when standards must be replaced.

b. Instrument Calibration and Tuning

Instrumentation requires calibration and tuning to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established detection limits. Each instrument is tuned and calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. The frequency of tuning, calibration, and the concentration of calibration standards is determined by the manufacturer's guidelines, the analytical method, or special program requirements and should be specified in the RLAB Method. Established calibration criteria must be met before analysis of samples, or data must be qualified. (See SOP 2410.10 for guidelines for data qualification.)

It is recognized, for multi-analyte analyses where the analytical standards are purchased as mixtures containing the analytes at equal concentrations, that the analytes may have widely differing responses. In these cases, a multi-point calibration curve may contain additional (e.g. 6 or more) calibration levels such that, for analytes with low response, the lower point(s) in the curve can be dropped/removed or, for analytes with high response, the upper point(s) in the curve can be dropped/removed in order to obtain an acceptable calibration for all of the analytes in the calibration mix. This is an acceptable practice as long as care it taken to note the reporting limit (lower end of the calibrated range) and upper end of the calibrated range for each analyte. However, it is not acceptable to drop/remove points in the middle of the curve.

5. Complete Set of QC Checks

For each batch of no more than 20 field samples, a set of laboratory QC samples (as appropriate for the method) will be analyzed. Typically this means that a prep and/or analysis batch will contain, in addition to field samples, the following four QC check samples.

- Laboratory control sample
- Method blank
- Matrix spike
- Matrix spike duplicate

Again, the QC required from contract laboratories shall be that specified in the SOWs and Task Orders. Whenever possible, contract laboratories shall be required to follow the procedures and meet the standards listed in this SOP. Data generated under the CLP Contract shall be evaluated in accordance with the SOWs and SOPs 2430.2, 2430.3, and 2430.4. Data generated under the REAP Contract shall be evaluated for adherence to the QA/QC requirements of the related Task Order SOW, which are based on the data quality objectives of the project.

a. Laboratory Control Sample (LCS)

An LCS is used to monitor the laboratory's batch-level performance of routine analytical methods. An LCS consists of a control matrix which has been spiked with a group of target compounds representative of the method analytes. The source of LCS spike solutions are kept as independent as possible from the calibration standards. A Performance Evaluation sample, obtained from an outside source, may be used for an

LCS. An LCS is analyzed with environmental samples to provide evidence that the laboratory is performing the analytical method within accepted QC guidelines.

For analyses with an extensive list of target analytes, not all analytes need be spiked. However, each LCS must include a representative mix of analytes and the laboratory shall ensure that all analytes in routine analyses are spiked within a two year period. For analyses having 1 to 10 analytes, spike all analytes. For analyses having 11 to 20 analytes, spike at least 10 or 80% whichever is greater. For analyses having more than 20 analytes, spike at least 16 analytes. Where the analysis contains multi-component analytes that interfere with accurate analysis of the mixture (e.g. technical chlordane, toxaphene, or PCBs), the LCS should contain those analytes that are most likely to be found in the samples and that don't interfere with one another.

Bias (percent recovery) data from the LCS are compared to control limits that have been established for each of the analytes monitored in the LCS. Control limits for bias are based on the historical average percent recovery of the LCS plus or minus three standard deviations (see SOP #2410.15 for details about statistical calculations and control limits). Decisions concerning laboratory performance of the method are based on QC data generated from an LCS.

In some situations control limits calculated by applying the above rules could result in an upper control limit (UCL) that is less than 105%, and/or a lower control limit (LCL) that is greater than 95% or lower than 10%. In cases where the calculation would result in the UCL being less than 105%, the UCL shall be established at 105%. In cases where the calculation would result in the LCL being greater than 95%, the LCL shall be established at 95%. In cases where the calculation would result in the LCL being less than 10%, the LCL shall be established at 10%.

Analytical data that are generated along with an LCS which falls within the established control limits are judged to be in control. Data generated along with an LCS which falls outside of the control limits are considered suspect and require corrective action or the associated data must be qualified (see SOP 2410.10). Corrective action shall include examination of instrument performance, sample preparation and analysis information, and a determination as to whether re-analysis is warranted. If the analyst has any doubt about the appropriate corrective action, the supervisor should be consulted.

An LCS has been established for each routine analytical method. Reagent water is used as the control matrix for the analysis of aqueous samples. The LCS compounds are spiked into reagent water and carried through the appropriate steps of the analysis. A universal blank matrix does not exist for solid samples. Therefore, at present, water is used as the blank matrix for inorganic analyses, and sodium sulfate is generally used for organic analyses. The LCS for solid samples consists of the LCS compounds spiked into the blank matrix and carried through the appropriate steps of the analysis.

b. Method Blank (MB)

Method blanks (reagent blanks, analytical blanks, or preparation blanks) are analyzed to assess the level of contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, cleanup, and analysis. Ideally, the concentration of an analyte in the method blank is below the method detection limit for that analyte. If it is not at least less than the reporting limit, corrective action shall be carried out. Corrective action shall consist of an investigation into, and elimination of, the source of the contamination (if at all possible), and re-analysis or qualification of the data.

When data must be qualified, the following "blank rule" shall be applied. No positive sample results will be reported without qualification for a field sample unless the concentration of the subject analyte in the sample exceeds 10 times the concentration in any associated method blank having that analyte at or above than the reporting limit. In instances where more than one method blank is associated with a given sample, qualification shall be based upon a comparison with the associated blank having the highest concentration of a contaminant. Sample results must NOT be corrected by subtracting any blank value. See SOP 2410.10 for further guidelines on data qualification.

It should be noted that the method blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "10 times" criteria, such that a comparison of the total amount of

contamination is actually made. It should also be noted that such qualified data may not meet the needs of the project. In such cases, corrective action and re-analysis shall be initiated if possible and practical.

c. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Matrix spike and spike duplicate results are used to assess the effects of a sample matrix on the analytical data. A minimum of one matrix spike/matrix spike duplicate set will be analyzed per batch of field samples. If a batch of field samples exhibits more than one apparent "sub-type" of matrix (e.g. different color, particle size, viscosity, etc.), more than one matrix spike/matrix spike duplicate set should be analyzed, one for each "sub-type" of matrix.

An MS is an aliquot from an environmental sample to which known concentrations of the analytes of interest have been added. The MS is taken through the entire analytical procedure and the percent recovery of the analyte results are calculated. MS percent recovery data are evaluated against control limits to assess the effect of the sample matrix on the bias of the analysis.

For analyses with an extensive list of target analytes, not all analytes need be spiked. However, each MS must include a representative mix of analytes and the laboratory shall ensure that all analytes in routine analyses are spiked within a two year period. For analyses having 1 to 10 analytes, spike all analytes. For analyses having 11 to 20 analytes, spike at least 10 or 80% whichever is greater. For analyses having more than 20 analytes, spike at least 16 analytes. Where the analysis contains multi-component analytes that interfere with accurate analysis of the mixture (e.g. technical chlordane, toxaphene, or PCBs), the MS should contain those analytes that are most likely to be found in the samples and that don't interfere with one another.

An MSD is a second aliquot of the same environmental sample used for the MS to which known concentrations (identical to those used for the MS) of the same analytes of interest are added. The MSD is taken through the entire analytical procedure along with the MS. In addition to the assessment of the sample matrix on the bias of the analysis, as described above, the measured results of the MSD are compared to those of the MS to determine the precision of the analysis. Precision results are expressed as Relative Percent Difference (RPD) between the MS and the MSD results, unless otherwise specified in the RLAB Method. Such data are

evaluated against control limits to assess the effect of the sample matrix on the precision of the analysis. It should be noted that for analyses where it is difficult to spike the MS and MSD at the same concentration level, evaluation of precision becomes problematic.

Control limits for bias are based on the historical average percent recovery of the MS and MSD plus or minus three standard deviations. Control limits for precision are based on the historical average relative percent difference plus three standard deviations. (See SOP #2410.15 for details about statistical calculations and control limits.)

In some situations control limits calculated by applying the above rules could result in an upper control limit (UCL) that is less than 105%, and/or a lower control limit (LCL) that is greater than 95% or lower than 10%, or a precision control limit (PCL) that is less than 5%. In cases where the calculation would result in the UCL being less than 105%, the UCL shall be established at 105%. In cases where the calculation would result in the LCL being greater than 95%, the LCL shall be established at 95%. In cases where the calculation would result in the LCL being less than 10%, the LCL shall be established at 10%. In cases where the calculation would result in the PCL being less than 5%, the PCL shall be established at 5%.

6. Additional QC Checks

a. Laboratory Fortified Blanks (LFB)

An LFB is similar to an LCS except that the spiking material is typically the same material as used in the calibration standard. Some methods specify the analysis of LFB samples in addition to, or instead of, an LCS. In general, these samples are treated in a fashion nearly identical to an LCS. (See SOPs 2410.10 and 2410.15.)

b. Laboratory Duplicates (LD)

Some analyses are not amenable to matrix spike analysis. For these analyses, precision is typically determined by the analysis of lab duplicates. Some methods specify the analysis of lab duplicates. In general, lab duplicate precision is treated in a fashion similar to MS/MSD precision. (See SOPs 2410.10 and 2410.15.)

c. Surrogates

Surrogates are compounds (normally organic) which should have similar chemical behavior to the analytes of interest, but which are not normally found in environmental samples. Surrogates are not routinely used in inorganic analyses. Surrogates are added to environmental samples to monitor the effect of the matrix on the bias of the analysis. Surrogate data can also be used to evaluate the effects of sample preparation on individual samples. Surrogate results are expressed in terms of percent recovery.

The laboratory routinely adds surrogates to samples requiring GC/MS (VOA, BNA) or GC (pesticide) analysis. The surrogate recoveries are evaluated against control limits to assess the effects of the matrix on analyte recovery. See SOP 2410.10 for information on surrogate recovery evaluation and SOP 2410.15 for information on surrogate recovery and control limit calculations.

d. Method of Standard Additions (MSA)

MSA is the practice of adding a series of known amounts of an analyte to aliquots of an environmental sample. The fortified samples are then analyzed and the recovery of the analytes calculated. MSA is generally used with metals and some conventional analyses to determine and compensate for the effect of the sample matrix on the bias of the analyses. MSA is required when the specific method, SOP, or regulation requires it, or when matrix specific QC checks indicate the need. Details will be contained in the RLAB Method where MSA is required.

e. Field Duplicates, Blanks, and Spikes

Field duplicates, field blanks, and field spikes are field QC samples submitted to the laboratory along with a batch of environmental samples. The results of field QC samples are used by the Project Manager in the data validation process. RLAB does not assess field QC sample results against control limits or qualify environmental sample data based on the results of field QC samples, but rather, reports field QC sample results the same as the results from environmental samples.

7. Sample Preservation and Holding Times

Samples must be properly collected and preserved and they must be analyzed within specified holding times in order for acceptable analytical results to be

obtained. Specific requirements should be listed in each RLAB Method. Also, see SOP 2420.6, "Sample Container Selection, Preservation and Holding Times."

F. SPECIFIC QC REQUIREMENTS

Specific QC requirements are contained in individual RLAB Methods and/or this SOP. However, Attachments #1, #2, and #3 are tables which summarize the QC required by EPA regulations or that are specified by the analytical methods required by those regulations which might in some cases require additional QC to that required by this SOP. These tables are not comprehensive, but they do include those analyses performed routinely by the Region 7 Laboratory for which there are specific methods required by the regulations. These attachments are included for ease of reference. Analysts must assure that all program required laboratory QC is performed for each analytical procedure.

These tables are based upon 30 basic categories of QC procedures. These categories are loosely defined to accommodate variations between methods and are spelled out in Attachment #4. Attachment #5 is a list of the acronyms used in these tables and their definitions.

A given QC procedure is considered required if, under any circumstances, a referenced method listed it as required. Not all QC which is listed as required is required each time the analysis is performed. For instance, MSA is required only if matrix spike recoveries indicate the need. Further, a given QC procedure is considered optional if it is mentioned in the method as desirable, but not mentioned as required under any circumstances.

The National Primary Drinking Water Regulations (NPDWR) require all monitoring to be conducted according to specified analytical methods, by certified laboratories. The Region 7 Laboratory does not actually perform many regulatory drinking water sample analyses. However, the ability and certification to perform such analyses is being maintained as a service to deal with eventualities. All drinking water analyses shall be performed according to appropriate regulatory requirements.

The National Pollution Discharge Elimination System (NPDES) regulations require all analyses in support of NPDES permits to be conducted in accordance with specified analytical methods. RLAB Methods have been written which conform to these regulatory requirements.

The Resource Conservation and Recovery Act (RCRA) regulations require the use of specified analytical methods in certain situations. The methods published in "Methods For The Evaluation Of Solid And Hazardous Wastes," SW-846, must be used when analyzing samples:

1. to be used in the de-listing process
2. relating to a trial burn
3. of free liquids
4. for waste characteristics testing

Otherwise, any scientifically sound analytical method may be used in support of those regulations. When analyzing samples which fall into the categories listed above, SW-846 methods and QC requirements shall be followed (technically sound and defensible methods are acceptable for all other program areas).

G. REFERENCES

1. Region 7 SOP 2410.10, Analytical Data Submission Package Contents and Review

2. Region 7 SOP 2410.11, Analytical Proficiency Demonstration in the Region 7 Laboratory

3. Region 7 SOP 2410.15, Estimating and Documenting Data Quality

4. Region 7 SOP 2410.19, Significant Figures (Digits)

5. Region 7 SOP 2420.6, Sample Container Selection, Preservation and Holding Times

6. Region 7 SOP 2430.2, Review of Data Deliverables Packages from Contract Laboratories (Format, Procedures and Content)

7. Region 7 SOP 2430.3, Contract Laboratory Program Data Review Functional Guidelines for Evaluating Organic (VOA, BNA, Pesticide/PCB) Analytical Data

8. Region 7 SOP 2430.4, Contract Laboratory Program Data Validation Functional Guidelines for Evaluating Inorganic Analytical Data

9. 40 CFR Part 136 Appendix B, Definition and Procedure for the Determination of the Method Detection Limit

QC Procedures
Required by The Drinking Water Methods
(December 2003)

(Additional Categories Beginning On Page 3)

Method Name/Number (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Forml Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp.
#110.1/Color[A]		*					*	*							
#120.1/Conductivity[D]		*						*							
#140.1/Odor[A]	*	*													
#150.1/pH[A,D]		*						*							
#160.1/Total Dissolved Solids (TDS)[A]		*													
#170.1/Temperature[D]		*					*								
#180.1/Turbidity[F]		*					*	*							
#200.7A/Metals by ICP[A,B,C,D] (Al[A],Ba[C],Ca[D],Cr[C],Cu[A,D],Fe[A],Mn[A],Ni[B], Ag[A],Na[C],Zn[A])		*	*		*	o	*	*			o		*	*	*
#200.9/Antimony (Sb) by GFAA[B]		*	*		*	o	*	*			o		*	*	o
#200.9/Arsenic (As) by GFAA[E]		*	*		*	o	*	*			o		*	*	o
#200.9/Beryllium (Be) by GFAA[B]		*	*		*	o	*	*			o		*	*	o
#200.9/Cadmium (Cd) by GFAA[C]		*	*		*	o	*	*			o		*	*	o
#200.9/Lead (Pb) by GFAA[D]		*	*		*	o	*	*			o		*	*	o
#245.1/Mercury (Hg) by ACV-AA[C]		*	*		*	o	*	*					*	*	o
#200.9/Selenium (Se) by GFAA[C]		*	*		*	o	*	*			o		*	*	o
#200.9/Thallium (Tl) by GFAA[B]		*	*		*	o	*	*			o		*	*	o
#310.1/Alkalinity by Pot. Tit.[D]		*						*							
#325.3/Chloride by Color. Tit.[A]	*	*													
#335.4/Cyanide by Color.[B]		*	*		*		*	*					*	*	*

[A] - Secondary Regulation, not enforceable.
[B] - Phase V Regulation, promulgated 7-17-92
[C] - Phase II Regulation, promulgated 1-30-91 & 7-1-91
[D] - Lead/Copper Rule, promulgated 6-7-91
[E] - NIPDWR, promulgated 12-75 & 7-76
[F] - Coliform & Surface Water Treatment Rules, promulgated 6-29-89
o - Optional
* - Required

Method Name/Number (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Formi Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte- rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr- ogate Spike	13 Lab Spike	14 Mat- rix Spike	15 Q.C. Samp.
SM 4500-F C Fluoride by ISE[A]							*	*							
#353.2/Nitrate[C], Nitrite[C], and Nitrate+Nitrite[C] by ACR		*	*		*		*	*					*	*	*
#365.1/Ortho-Phosphate by Auto. Color.[D]							*	*							
#370.1/ Silica (Si) by Color.[D]							*	*							
#375.4/Sulfate by Auto.Turb.[A,B]							*	*							
SM16 408C/Free Chlorine by Amp. Tit.[F]															
SM16 512A/Foaming Agents by MBAS[A]							*	*							
#524.2/Volatile Organics by GC/MS		*	*		*	*	*	*	*	*		*	*	o	*
#504/EDB & DBCP by GC/ECD[C]		*	*	*	*	o	*						*		*
#507/Nitrogen & Phosphorus Pest. by GC/NPD[B,C]	*	*	*		*	*	*	*	o			*	*	*	*
#508/Chlorinated Pest. by GC/ECD[B,C]	*	*	*			o	*	*	o			*	*	*	*
#1613/Dioxin by GC/HRMS[B]	*	*	*	*		*	*	*	*	*		*	*	o	*
#515.1/Chlorinated Acid Herb. by GC/ECD[B,C]	*	*	*			*	*	*	o			*	*	*	*
#550.1/PAHs by HPLC[B]		*	*	*		*	*	*	o				*	*	*
Drinking Water Regulations		*			*										
Drinking Water Laboratory Certification Man.	*			*			*	*					*	*	*

* The Drinking Water Regulations also require laboratory certification, specific methods, acceptable performance evaluation (PE) study performance, and maximum detection limits.

* The Drinking Water Laboratory Certification Manual also requires chain-of-custody procedures, on-site audits, equipment and supplies specifications, records, and wavelength accuracy checks.

[A] - Secondary Regulation, not enforceable.
[B] - Phase V Regulation, promulgated 7-17-92
[C] - Phase II Regulation, promulgated 1-30-91 & 7-1-91
[D] - Lead/Copper Rule, promulgated 6-7-91
[E] - NIPDWR, promulgated 12-75 & 7-76
[F] - Coliform & Surface Water Treatment Rules, promulgated 6-29-89
o - Optional
* - Required

QC Procedures
Required by The Drinking Water Methods
(December 2003)

(Continued From Previous Pages)

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup.	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entiv Maint.	29 Serv. Cont-ract	30 Bal. Check
#110.1/Color[A]	*														
#120.1/Conductivity[D]													*		
#140.1/Odor[A]															
#150.1/pH[A,D]													*		
#160.1/Total Dissolved Solids (TDS)[A]															
#170.1/Temperature[D]															
#180.1/Turbidity[F]	*														
#200.7A/Metals by ICP[A,B,C,D] (Al[A],Ba[C],Ca[D],Cr[C],Cu[A,D],Fe[A],Mn[A],Ni[B], Ag[A],Na[C],Zn[A])	*						o	*			*				
#200.9/Antimony (Sb) by GFAA[B]	*		o				o	*			*			o	o
#200.9/Arsenic (As) by GFAA[E]	*		o				o	*			*			o	o
#200.9/Beryllium (Be) by GFAA[B]	*		o				o	*			*			o	o
#200.9/Cadmium (Cd) By GFAA[C]	*		o				o	*			*			o	o
#200.9/Lead (Pb) by GFAA[D]	*		o				o	*			*			o	o
#245.1/Mercury (Hg) by ACV-AA[C]	*										*	*			
#200.9/Selenium (Se) by GFAA[C]	*		o				o	*			*			o	o
#200.9/Thallium (Tl) by GFAA[B]	*		o				o	*			*			o	o
#310.1/Alkalinity by Pot. Tit.[D]															
#325.3/Chloride by Color. Tit.[A]															
#335.4/Cyanide by Color.[B]	*										*				

[A] - Secondary Regulation, not enforceable.
[B] - Phase V Regulation, promulgated 7-17-92
[C] - Phase II Regulation, promulgated 1-30-91 & 7-1-91
[D] - Lead/Copper Rule, promulgated 6-7-91
[E] - NIPDWR, promulgated 12-75 & 7-76
[F] - Coliform & Surface Water Treatment Rules, promulgated 6-29-89
o - Optional
* - Required

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup.	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entiv Maint.	29 Serv. Cont-ract	30 Bal. Check
SM 4500-F C Fluoride by ISE[A]	*														
#353.2/Nitrate[C], Nitrite[C], and Nitrate+Nitrite[C] by ACR	*										*				
#365.1/Ortho-Phosphate by Auto Color.[D]	*														
#370.1/Silica (Si) by Color.[D]	*														
#375.4/Sulfate by Auto Turb.[A,B]	*														
SM16 408C/Free Chlorine by Amp. Tit.[F]													*		
SM16 512A/Foaming Agents by MBAS[A]	*														
#524.2/Volatile Organics by GC/MS	*	o			*										
#504/EDB & DBCP by GC/ECD[C]	*	o		o	o						*				
#507/Nitrogen and Phosphorus Pest. by GC/NPD[B,C]	*	o	o	o	*					*	*				
#508/Chlorinated Pest. by GC/ECD[B,C]	*	o	o	o	*	*				*	*				
#1613/Dioxin by GC/HRMS[B]	*			o	*				*	*					
#515.1/Chlorinated Acid Herb. by GC/ECD[B,C]	*	o	o	o	*					*	*				
#550.1/PAHs by HPLC[B]	*	o	o	o	o				*						
Drinking Water Regulations															
Drinking Water Laboratory Certification Manual	*												*		*

[A] - Secondary Regulation, not enforceable.
[B] - Phase V Regulation, promulgated 7-17-92
[C] - Phase II Regulation, promulgated 1-30-91 & 7-1-91
[D] - Lead/Copper Rule, promulgated 6-7-91
[E] - NIPDWR, promulgated 12-75 & 7-76
[F] - Coliform & Surface Water Treatment Rules, promulgated 6-29-89
o - Optional
* - Required

QC Procedures
Required by The NPDES Wastewater Methods
(December 2003)

(Additional Categories beginning on Page 4)

Method Number/Name (Analytes)	1 Anal. Qual.	2 Samp.. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Formal Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp.
INORGANICS -															
#160.1[A]/SM16 #209B - TDS		*													
#160.2[A]/SM16 #209C - TSS		*													
#160.3[A]/SM16 #209A - Total Solids		*													
#200.7[A] - ICP Metals (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Ca, Mg, Na, K)		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Antimony (Sb) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Arsenic (As) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Cadmium (Cd) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Chromium (Cr) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Lead (Pb) by GFAA		*	*		*	o	*	*			o		*	*	o
#245.1[A] - Mercury (Hg) by ACV-AA		*	*		*	o	*	*					*	*	o
#200.9[A&B] - Selenium (Se) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Silver (Ag) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Thallium (Tl) by GFAA		*	*		*	o	*	*			o		*	*	o
#335.3[A] - Cyanide, Total		*					*	*					*	*	o
#350.1[A] - Ammonia		*	*		*		*						*	*	*
#351.2[A] - TKN		*					*								
#353.2[A] - Nitrate + Nitrite		*					*								

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

Method Number/Name (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Formal Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Internal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surrogate Spike	13 Lab Spike	14 Matrix Spike	15 Q.C. Samp.
#365.1[A] - OrthoPhosphate		*	*		*		*	o					*	*	*
#365.4[A] - Total Phosphorus		*					*								
9036[C&D] - Sulfate		*					*								
#376.2[A] & SM16 #427C - Sulfide		*					*								
#410.1[A] - COD		*					*								
EPA 1664 - Oil and Grease		*	*		*								*	*	
#420.2[A] - Phenolics (4AAP)		*					*								
SM 2510 B - Spec. Conductivity							*	*							
SM16 #209D - Volatile Solids		*													
SM16 #214A - Turbidity		*					*	*							
SM14 #307B - Hexavalant Chromium		*					*								
SM16 #314A - Hardness (by calculation)	—	None	—												
SM16 #402 - Acidity		*													
#310.1[A] - Alkalinity		*													
SM 4500-Cl B - Chloride		*					*								
SM16 #412F - Cyanide Amenable to Chlor.		*					*								
SM 4500-F C - Fluoride		*					*	*							
SM16 #421B - Dissolved Oxygen		*													
EPA 150.1 - pH		*					*								
SM16 #505B - TOC		*					*								
SM 5210 B - BOD		*					*						*		
SM16 #512B - Surfactants (MBAS)							*								

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

Method Number/Name (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Forml Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp.
ORGANICS -															
#608[E] - Pesticides and PCBs	*	*	*	*	*	*	*	*	o				o	*	*
#624[E] - VOCs	*	*	*	*	*	*	*	*	*	*		*		*	*
#625[E] - SemiVolatiles	*	*	*	*	*	*	*	*	*	*		*		*	*
Regulations -		*			*										

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
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[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

QC Procedures
Required by The NPDES WasteWater Methods
(December 2003)

(Continued From Previous Pages)

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup./ Rep. Spike	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entiv Maint.	29 Serv. Cont-ract	30 Bal. Check
INORGANICS -															
#160.1[A]/SM16 #209B - TDS															
#160.2[A]/SM16 #209C - TSS															
#160.3[A]/SM16 #209A - Total Solids															
#200.7[A] - ICP Metals (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Ca, Mg, Na, K)	*						o	*			*				
#200.9[A&B] - Antimony (Sb) by GFAA	*						o	*			*		*		
#200.9[A&B] - Arsenic (As) by GFAA	*						o	*			*		*		
#200.9[A&B] - Cadmium (Cd) by GFAA	*						o	*			*		*		
#200.9[A&B] - Chromium (Cr) by GFAA	*						o	*			*		*		
#200.9[A&B] - Lead (Pb) by GFAA	*						o	*			*		*		
#245.1[A] - Mercury (Hg) by ACV-AA	*											*	*		
#200.9[A&B] - Selenium (Se) by GFAA	*						o	*			*		*		
#200.9[A&B] - Silver (Ag) by GFAA	*						o	*			*		*		
#200.9[A&B] - Thallium (Tl) by GFAA	*						o	*			*		*		
#335.3[A] - Cyanide, Total	*														
#350.1[A] - Ammonia	*														
#351.2[A] - TKN	*														
#353.2[A] - Nitrate + Nitrite	*														

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
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* - Required

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup./ Rep. Spike	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entiv Maint.	29 Serv. Cont-ract	30 Bal. Check
#365.1[A] - OrthoPhosphate	*														
#365.4[A] - Total Phosphorus	*														
9036[C&D] - Sulfate	*														
#376.2[A] & SM16 #427C - Sulfide	*														
#410.1[A] - COD	*														
EPA 1664 - Oil and Grease	*														
#420.2[A] - Phenolics (4AAP)	*														
SM 2510 B - Spec. Conductivity															
SM16 #209D - Volatile Solids															
SM16 #214A - Turbidity															
SM14 #307B - Hexavalant Chromium	*														
SM16 #314A - Hardness (by calculation)	—	None	—												
SM16 #402 - Acidity															
#310.1[A] - Alkalinity															
SM 4500-Cl B - Chloride															
SM16 #412F - Cyanide Amenable to Chlor.	*														
SM 4500-F C - Fluoride															
SM16 #421B - Dissolved Oxygen															
EPA 150.1 - pH															
SM16 #505B - TOC	*														
SM 5210 B - BOD	*														
SM16 #512B - Surfactants (MBAS)	*														

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup./ Rep. Spike	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entiv Maint.	29 Serv. Cont-ract	30 Bal. Check
ORGANICS -															
#608[E] - Pesticides and PCBs	*		o	o	o				*						
#624[E] - VOCs	*		o	o											
#625[E] - SemiVolatiles	*		o	o						*					
Regulations -															

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

QC Procedures

Required by The RCRA Solid and Hazardous Waste Methods

(December 2003)

(Additional Categories On Page 2)

Method Number/Name (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Forml Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp
#1020[A]/Flash Point		*					*								
#1311[A]/TCLP	*	*		*	*	*								*	*
#6010B[A]/ICP Metals (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Ti[B], Tl, V, Zn, Ca, Mg, Na, K)	*	*		*	*	*	*	*			*		*	*	*
#7041[A&C]/Antimony (Sb) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7060[A&C]/Arsenic (As) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7131[A&C]/Cadmium (Cd) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7191[A&C]/Chromium (Cr) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7196[A]/Hexavalant Chromium (Cr^{+6})		*					*	*			*		*	*	
#7421[A&C]/Lead (Pb) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7471[A]/Mercury (Hg) by ACV-AA	*	*		*	*	*	*	*			*		*	*	*
#7740[A&C]/Selenium (Se) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7761[A&C]/Silver (Ag) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7841[A&C]/Thallium (Tl) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#8081A[A&D]/Pesticides and #8082[A&D]/PCBs	*	*	*	*	*	*	*	*	o	*		*	*	*	*
#8151[A]/Herbicides	*	*		*		*	*	*	o		o	*		*	*
#8260[A]/Volatile Organics	*	*		*		*	*	*	*	*	o	*		*	*
#8270[A]/Semi-Volatile Organics	*	*		*		*	*	*	*	*	o	*		*	*
#8290[A]/Dioxin	*	*		*			*	*	*	*			*	o	o
#9010B[A]/Cyanide	*	*		*	*	*	*	*			*		*	*	*
Regulations -	----	None	----												

[A] - SW-846, "Methods for the Evaluation of Solid and Hazardous Waste"
[B] - This analyte is not listed in the SW-846 ICP method.
[C] - The general AA methods section, #7000, is also referenced.
[D] - Refers to other SW-846 Methods
[E] - Applies to 8081A only
o - Optional
* - Required

Attachment #3

QC Procedures

Required by The RCRA Solid and Hazardous Waste Methods

(December 2003)

(Continued From Previous Page)

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup./ Rep. Spike	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entive Maint.	29 Serv. Cont-ract	30 Bal. Check
#1020[A]/Flash Point			*										*		
#1311[A]/TCLP	*		*										*		*
#1610B[A]/ICP Metals (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Sc, Ti[B], Tl, V, Zn, Ca, Mg, Na, K)	*		*				*	*					*		*
#7041[A&C]/Antimony (Sb) by GFAA	*		*				*	*					*		*
#7060[A&C]/Arsenic (As) by GFAA	*		*				*	*					*		*
#7131[A&C]/Cadmium (Cd) by GFAA	*		*				*	*					*		*
#7191[A&C]/Chromium (Cr) by GFAA	*		*				*	*					*		*
#7196[A]/Hexavalant Chromium (Cr^{+6})	*		*												
#7421[A&C]/Lead (Pb) by GFAA	*		*				*	*					*		*
#7471[A]/Mercury (Hg) by ACV-AA	*		*				*					*	*		*
#7740[A&C]/Selenium (Se) by GFAA	*		*				*	*					*		*
#7761[A&C]/Silver (Ag) by GFAA	*		*				*	*					*		*
#7841[A&C]/Thallium (Tl) by GFAA	*		*				*	*					*		*
#8081A[A&D]/Pesticides and #8082[A&D]/PCBs	*		*		o	* [E]			*				*		
#8151[A]/Herbicides	*	o	*		o				*						
#8260[A]/Volatile Organics	*	*	*		*						*				
#8270[A]/Semi-Volatile Organics	*	o	*		*	o				o	*				
#8290[A]/Dioxin		o	o						o	*					
#9010B[A]/Cyanide	*	*	*	*									*		*
Regulations -	----	None	----												

[A] - SW-846, "Methods for the Evaluation of Solid and Hazardous Waste"
[B] - This analyte is not listed in the SW-846 ICP method.
[C] - The general AA methods section, #7000, is also referenced.
[D] - Refers to other SW-846 Methods
[E] - Applies to 8081A only
o - Optional
* - Required

Attachment #3

Category Codes

The following categories represent requirements mandated in methods:

1 - Analyst Qualifications
2 - Sample Collection, Preservation and Holding Procedures
3 - Procedures for Initial Demonstration of Ability with the Method
4 - Formal Quality Assurance/Quality Control (QA/QC) Program
5 - Method Detection Limit (MDL) Determination
6 - Quality Control (QC) Charts and Statistics
7 - Initial Calibration Procedures
8 - Continuing Calibration Procedures
9 - Internal Standard Procedures
10 - Mass Spectrometer (MS) Tuning Procedures
11 - Method of Standard Additions (MSA)
12 - Surrogate Spikes (samples fortified with surrogate compounds)
13 - Laboratory Spikes (fortified blanks) (LFB or LCS)
14 - Matrix Spikes (fortified sample) (MS/MSD)
15 - Quality Control Samples (non-blind sample from an external source) (LCS or PE)
16 - Reagent Blanks (MB)
17 - Field Blank (blank sample taken to the field and shipped with the samples) (FB)
18 - Laboratory Duplicate (re-analysis of a sample split in the laboratory) (LD)
19 - Field Duplicate (two collocated samples or one sample split into two in the field) (FD)
20 - Qualitative Confirmation (re-analysis by another technique to confirm the analyte identity)
21 - Analyte Break-Down Check (to identify if any analytes are becoming other compounds during analysis)
22 - Serial Dilution (to identify and/or eliminate matrix interferences)
23 - Interference Check Samples (to identify ICP inter-element interferences)
24 - Clean-up Validation (to verify recovery of analytes from sample clean-up procedures)
25 - Gas Chromatographic Column Performance Check (to verify acceptable chromatography)
26 - Instrument Performance Check (to verify acceptable instrument performance during a given analytical run)
27 - Flow Rate Check
28 - Preventive Maintenance Procedures
29 - Service Contracts
30 - Balance Check (to verify the proper working of the analytical balance)

Acronyms

AA - Atomic Absorption
ACR - Automated Cadmium Reduction
ACV - Automated Cold Vapor
Amp. Tit. - Amperometric Titration
Auto. Color. - Automated Colorimetric
Auto. Turb. - Automated Turbidimetric

BOD - Biochemical Oxygen Demand

CFR - Code of Federal Regulations
Chlor. - Chlorination
COD - Chemical Oxygen Demand
Color. - Colorimetric
Color. Tit. - Colorimetric Titration
(cont.) - Continued from previous pages

DBCP - DiBromoChloroPropane

ECD - Electron Capture Detector
EDB - Ethylene DiBromide
ELCD - Electrolytic Conductivity Detector

GC - Gas Chromatography
GFAA - Graphite Furnace Atomic Absorption

Herb. - Herbicide
HRMS - High Resolution Mass Spectrometer (used as a GC detector)
HPLC - High Performance Liquid Chromatography

ICP - Inductively Coupled Plasma
ISE - Ion Selective Electrode

MBAS - Methylene Blue Active Substances
MDL - Method Detection Limit
MSA - Method of Standard Additions

NPD - Nitrogen Phosphorus Detector
NPDES - National Pollution Discharge Elimination System

PCBs - PolyChlorinated Biphenyls
Pest. - Pesticide
PID - Photo Ionization Detector
Pot. Tit. - Potentiometric Titration

QA - Quality Assurance
QC - Quality Control

RCRA - Resource Conservation Recovery Act

SM14 - Standard Methods for the Analysis of Water and Waste, 14th ed.
SM16 - Standard Methods for the Analysis of Water and Waste, 16th ed.
Spec. - Specific

TCLP - Toxicity Characteristic Leachate Procedure
TKN - Total Kjeldahl Nitrogen
TOC - Total Organic Carbon
Trmt. - Treatment

VOCs - Volatile Organic Compounds

4AAP - 4-Amino-AntiPyrine

STANDARD OPERATING PROCEDURE

No. 2410.1E

ANALYTICAL DATA MANAGEMENT PROCEDURES

August 25, 2005

Delores Simmons
ENSV/RLAB/CATS

APPROVED:

Nicole Rowley — Peer Reviewer — 8/30/05 — Date

Laboratory Manager — 8/31/05 — Date

Harold D. Brown — Independent QA Reviewer — 9/01/05 — Date

Recertified

Reviewer					
Date					

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A. PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a uniform policy and process for the generation, transmittal, storage, and retrieval of information within the Regional Laboratory (RLAB), Environmental Services Division (ENSV).

B. APPLICABILITY

These procedures are applicable to all RLAB personnel and to contractors providing analytical support to the RLAB.

C. SUMMARY OF PROCEDURES

It is the ENSV policy that all Divisional activities in support of the investigation and enforcement of environmental activities are conducted in a documented, uniform, thorough, and professional manner conducive to the generation, availability, and use of environmental data of known and documented quality. The specific procedures discussed below, and in more detail in the referenced SOPs, are intended to permit the systematic achievement of this policy goal when implemented as routine practice.

For the purposes of this SOP, "information" is defined to include both sample-related data (sampling time, date, and location; analytical results) and documentation related to the generation and processing of sample data. Items in the latter category include Quality Assurance Project Plans (QAPPs) request forms, tracking documents, and data review. Given the broad purpose of this SOP, information for specific tasks may be presented as an overview. When this occurs, reference is made to an appropriate SOP for more detailed discussions.

D. DEFINITIONS/ACRONYMS

Activity	Sampling effort
ASR	Analytical Services Request
CATS	Contractor and Technical Support Section
CLP	Contract Laboratory Program
ENSV	Environmental Services Division
EPA	Environmental Protection Agency
LAN	Local Area Network
LIMS	Laboratory Information Management System
PM	Project Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control

RCRA	Resource Conservation and Recovery Act
REAP	Regional Environmental Analysis Program
RLAB	Regional Laboratory Branch (commonly referred to as the Regional Laboratory)
RSCC	Regional Sample Control Coordinator
RSTC	Regional Science and Technology Center (facility where the RLAB is located)
SOP	Standard Operating Procedure

E. PERSONNEL QUALIFICATIONS

Personnel should be knowledgeable in data management and demonstrate organizational and communication skills.

F. PROCEDURES FOR INFORMATION GENERATION AND FLOW

1. The procedures covered by this SOP are designed to address the management of the following types of information and information flow processes:

   - Analytical Services Request (ASR) Form
   - Sample collection, preservation, analysis, and storage documentation
   - Data review and validation documentation
   - Data entry and storage in the Laboratory Information Management System (LIMS) computer data management and reporting system
   - Distribution of sampling documentation and sample data to project leaders
   - Retrieval of sample data from storage

2. Presampling Activities

   a. After approval of the QAPP, presampling activities begin. Presampling activities require actions by both the field sampling group and RLAB personnel to prepare for the arrival of samples. Based on the ASR form submitted, the RLAB Manager assigns analytical resources to the ASR, based on the laboratory's capabilities and workload.

   b. The assignment of analytical resources may also include the tracking and storage of data in the LIMS system. All analyses to be conducted are entered into the LIMS system; the laboratory is not authorized to perform analyses other than those requested.

c. The LIMS system is also used to generate field sheets (for data collection in the field) and sample tags (to identify sample containers) at this time. Procedures for obtaining field sheets and sample tags are contained in SOP No. 2420.13.

d. Prior to sampling, the sampler(s) pick up the field sheets, sample tags, and any other requested containers or field blanks.

3. Sample Management/Post Sampling Activities

a. After collecting samples and field data, ship the samples and associated completed paperwork (i.e., chain-of-custody and field sheets) to the STC.

b. The Regional Sample Control Coordinator (RSCC) (or backup) receives the samples as described in SOP 2420.1.

c. The RSCC (or backup) logs the samples into the LIMS system, enters field information contained on the field sheets (i.e., temperature or pH), sends out sample receipt notification via the Local Area Network (LAN), and performs any editing necessary to correct the requested parameters defined in LIMS, when applicable.

d. The RSCC (or backup) coordinates in-house, as well as Contract Laboratory Program/ Regional Environmental Analysis Program (CLP/REAP) shipments, and transfers all completed shipping and sampling documentation to the Data Coordinator.

e. Prepare and analyze samples according to the approved STC analytical methods. During sample analyses, a large amount of QC data is generated using EPA analytical protocol. These data are reviewed by the analyst(s) and his/her supervisor to assess data quality and decide if re-analyses are needed. Generate and proofread appropriate data reporting forms, as described in SOP No. 2410.10. Submit the completed and reviewed data package to the Manager, CATS, who date stamps and forwards it to the Data Coordinator.

4. Data Transmission and Storage Procedures

a. Primary Activity File and Back-Up File

RLAB maintains a hard copy of all in-house and contractor data for each ASR in primary folders and back-up files. SOPs 2410.2 and 2410.10 contain a complete description of the content of these files and the procedures for their management. These are the official records for sampling/analytical ASRs.

Back-up files contain all supporting laboratory raw data such as sample preparation and instrumental information generated during the analysis of the samples.

b. Transmission of Analytical Data

Whenever an ASR is complete, the RLAB Manager approves the transmittal of the analytical data to the Project Manager (PM). The transmittal consists of field sheets, chain-of-custody, memoranda concerning the ASR, and a LIMS Analysis Requested Report.

Also, an electronic transmittal is sent to the PM via e-mail. This electronic transmission consists of an identical LIMS Analysis Request Report and the data in spreadsheet format.

c. Storage of Laboratory Data

Laboratory data contained in the primary and back-up files are maintained by the Data Coordinator and the CATS Section according to procedures described in SOPs 2410.2 and 2410.3.

5. Data Retrieval From Long-Term Storage

In the event a hard copy of a data package in storage is required, the Data Coordinator or the CATS Program Manager is to be notified. The data will then be retrieved according to procedures described in SOPs 2410.2 and 2410.3.

G. RECORDS MANAGEMENT

The procedures covered by this SOP are designed to address the management by the CATS section of the following types of information and information flow processes:

- Analytical Services Request (ASR) Form
- Sample collection, preservation, analysis, and storage documentation
- Data review and validation documentation
- Data entry and storage in the Laboratory Information Management System (LIMS) computer data management and reporting system
- Distribution of sampling documentation and sample data to project managers
- Retrieval of sample data from storage.

H. QUALITY ASSURANCE/QUALITY CONTROL

Upon completion of an analytical assignment and after the peer reviewer is satisfied with the package, it is submitted for further review as stated in SOP 2440.5, "U.S. EPA Region 7 Laboratory Quality Assurance Project Plan," Section 9, and reviewed for compliance as stated in SOP 2430.6, "Periodic Internal Program Review of the Region 7 Laboratory," Section D.3.b.10. Once all of the requested analyses have been completed, the Data Coordinator reviews the analytical data files and assures that all needed documentation is included. Any missing information is obtained from the analysts prior to data transmission to the Project Manager.

I. REFERENCES

1. US EPA, Region 7, "Analytical Data Files," Environmental Services Division Operations and Quality Assurance Manual, SOP 2410.2

2. US EPA, Region 7, "Long-Term Analytical Records Management," Environmental Services Division Operations and Quality Assurance Manual, SOP 2410.3

3. US EPA, Region 7, "Procedures For Preparation Of Field Sheets And Tags For RCRA And Superfund Activities," Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.13

4. US EPA, Region 7, "Sample Receipt And Log-In," Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.1

5. US EPA, Region 7, "Analytical Data Submission Packages," Environmental Services Division Operations and Quality Assurance Manual, SOP 2410.10

6. US EPA, Region 7, "Periodic Internal Program Review Of The Region 7 Laboratory," Environmental Services Division Operations and Quality Assurance Manual, SOP 2430.6

7. US EPA, Region 7, "U.S. EPA Region 7 Laboratory Quality Assurance Operating Plan," Environmental Services Division Operations and Quality Assurance Manual, SOP 2440.5